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CHEMICAL AGE

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DCL LP POLYTHENE

ALTHOUGH Distillers Co. Ltd. have a licence for the Phillips low-pressure process for polythene, and, moreover, have, through their part owned subsidiary British Hydrocarbon Chemicals Ltd., a plant in the course of erection to produce polythene by the Phillips' process, they also have their own low-pressure process.

News of this DCL process is revealed in the complete patent specification published this month (BP. 800 783) and granted to H. Beauchamp and A. L. J. Raum for Distillers. The complete specification was filed on 24 September 1956 and the application date was 25 October 1955 (No. 30 508/55).

The patent gives details of a process for the polymerisation of ethylene to give polymers having high molecular weights which are suitable for the preparation of films, fibres, moulded articles and the like. In particular, it relates to the production of high molecular weight polymers under 'mild conditions of temperature and pressure'.

According to this DCL patent the process comprises contacting ethylene with a catalyst system formed by mixing a hydrocarbon cadmium compound free from ethylenic unsaturation with a titanium halide.

More specifically any hydrocarbon cadmium compound free from ethylenic unsaturation can be used to form the catalyst system. Such compounds are those represented by the formula $Cd R' R''$ where R' and R'' are the same or different hydrocarbon radicals, as for example, alkyl, aralkyl, alkaryl and cycloaliphatic radicals. Specific examples quoted in the patent are cadmium di-methyl, cadmium di-ethyl, cadmium di-*n*-propyl and cadmium diphenyl. Of the preferred compounds, cadmium di-ethyl is one, since it is a liquid and its use in the process is stated to be 'particularly advantageous'.

Any of the halides of titanium can be used, the patent indicates, together with the cadmium compound, to form the catalyst system. In this case, the preferred compounds are stated to be titanium tetrachloride, titanium trichloride and titanium dichloride. Since titanium tetrachloride is a covalent liquid which is readily available commercially, it is the titanium preparation of choice. The catalyst system should be formed from a molar excess of the hydrocarbon cadmium compound over the titanium present. Preferably at least two molar proportions of the hydrocarbon cadmium compound are employed to each proportion of the titanium halide, particularly if the tetravalent titanium is used. When lower halides of titanium are used, lower molar proportions of the hydrocarbon cadmium can be used.

Preparation of the catalyst systems and the subsequent polymerisation reaction should be effected in the complete absence of molecular oxygen, carbon dioxide and water, the patent records. A suitable atmosphere is ethylene. An inert gas, for example, nitrogen can be used to flush out the polymerisation vessel prior to admission of the various components of the reaction mixture. Oxygen, carbon dioxide or water destroys the catalyst systems or their components and little or no polymerisation will occur.

In the DCL process, polymerisation is effected with the components of the reaction dispersed throughout an inert liquid vehicle. This vehicle is,

for preference, a solvent for at least one of the compounds which react together to form the catalyst system and for the ethylene. Solvents mentioned in the patent are aliphatic, cycloaliphatic and hydrogenated aromatic hydrocarbons such as pentane, hexane, cyclohexane, tetrahydronaphthalene and decahydronaphthalene. Other suitable liquids mentioned are benzene, and xylene, and halogenated aromatic hydrocarbons (e.g. ortho-dichlorobenzene and chlorinated naphthalene). The hydrocarbon cadmium compound, it is stated, is often most conveniently prepared in an ether solution, but all the ether should be removed from the cadmium compound before it is used to form the catalyst system. Either the two components are mixed in an inert liquid vehicle to form the catalyst system and the ethylene is run in or, the components of the system are mixed in the presence of ethylene. In this case, rapid polymerisation of ethylene takes place, so that further quantities of ethylene may be added to the reaction mixture and polymerised.

It is stated that the catalyst systems described in the present patent are generally sufficiently active for polymerisation to be initiated at normal ambient temperature or below, for instance, 10°C. Raising the temperature generally increases the rate of polymerisation. Normally, however, it is undesirable to employ temperatures in excess of 150°C. Elevated pressures are not required to bring about the polymerisation of ethylene. Nevertheless, for convenience of handling gaseous ethylene, it is indicated that it is convenient to use slightly elevated pressures. For preference, the DCL process is carried out with the ethylene under a pressure in the range 50 to 500 p.s.i.g.

Conventional techniques are used to recover the high molecular weight polythene produced by this DCL process and to work it into its final form. Use of a mineral acid washing stage is recommended during the working up process to remove metallic contaminants. The process can be used batch-wise or continuously, and good yields of high grade polythene are claimed. The interest in this DCL patent lies in its close resemblance to the Ziegler processes for low-pressure polythenes, making use as it does of hydrocarbon metallic compound and titanium-halide for the catalyst system and the use of low pressures (50 to 500 p.s.i.g.). The question is, are Distillers likely to use their own process, or will British Hydrocarbon Chemicals take it up? Or have Distillers, aware of the Ziegler and Phillips processes and the difficulties and expense of working up the process on a commercial scale, decided that it is more economic to produce low-pressure polythene by the Phillips process? Phillips, of course, have worked their process on a commercial scale and in their licensing arrangements provide all engineering design, etc. Ziegler, on the other hand, in licensing his process, allows the use of his method. Commercial scale details of process plant, etc., have to be worked out by the company taking up the licence.

US POLYPROPYLENE NEWS

SINCE its introduction in the US in late 1957 by Hercules Powder, polypropylene has been in good demand. Recently Jersey Standard's affiliate, Humble Oil, announced plans to produce polypropylene at their Baytown, Texas, unit by early 1960. Initial output will be 40 million lb. a year. Standard's Enjay affiliate will join Spencer Chemical in a four-year polypropylene resale agreement.

Until the Humble plant comes into production, Spencer Chemical are to supply development quantities from Jersey Standard's one-ton-a-day pilot plant, and from Enjay. This agreement gains for Standard and their affiliates the advantage of Spencer Chemicals plastics marketing experience.

Other US companies known to be interested in polypropylene include Eastman, Monsanto and Dow. Eastman

are in fact making the polymer at their Longview, Texas, pilot facilities. The company has plans for a commercial plant, but has not yet indicated when this will be operating. A Tennessee Eastman process is being used and the company has applied for several catalyst patents.

Hercules Powder are to follow their May price reduction of polypropylene by a further cut of 7 cents to 49 cents per lb. for their natural coloured moulding powder. The price of the coloured resin has also been dropped from 65 to 58 cents per lb. At the same time, Hercules are introducing two new grades of polypropylene.

Hercules, who have a 20 million lb. capacity plant at Parlin, New Jersey, are understood to have made plans to expand their polypropylene production.

With the exception of the first producers of polypropylene, Montecatini of Italy and Farbwerke Hoechst-AG no other European plastics producer has as yet indicated any plans for production of the polymer.

WASTE DISPOSAL PROCESS

A COMMERCIAL unit for waste disposal is now in operation in Chicago, US, and another will start up later this year. The unit makes use of a new continuous method for wet oxidation of wastes. In this process, which has been developed by Salvo Chemical Corporation, wastes are burned with water pressure as completely, it is claimed, as the usual method of first evaporating the water and then incinerating the dried residue. End products in each case are steam, nitrogen, carbon dioxide and ash.

While consumption of aqueous chemical, paper mill and sewage waste liquors is effected, operating and excess power and steam are generated. An odourless, low oxygen demand effluent is discharged. This wet oxidation of wastes, in fact, provides an economic source of power and chemical or energy recovery from cheap fuel sources.

In the process, air is mixed with waste liquor in the reactor, and temperature is anywhere below 705°F. Typical reactor conditions are 500 to 600°F and 1,100 to 2,000 p.s.i.g. The reactor products move forward continuously. Process steam can be formed by the products giving up excess heat in an exchanger. Liquid and gas phases are separated, the water phase containing the ash.

The liquid phase passes countercurrently to the incoming air in a humidifier, where the air is heated and the oxidised effluent liquor is cooled. Reactor gases become saturated with steam by contact with water in the reactor. These gases containing up to 20 per cent CO₂ may be superheated before expansion through a gas turbine which supplies power for air compressor and liquor pumps.

The process operates with only enough air that oxygen is just detected by the stack gas analysers. As an example, a waste sulphate liquor containing 140 gm./litre chemical oxygen demand requires 5 lb. air/gall. waste to completely oxidise the fuel and the combustion liberates 6,830 B.Th.U./gall. waste.

With regard to chemical recovery, spent liquor from a paper mill contains sodium salts of lignin sulphonic acid. After the liquor is passed through the wet oxidation unit, lignin sulphonic acid is oxidised to sodium sulphate, which can be converted to sodium sulphite for re-use in the paper mill.

The unit installed in the US is that of the Southwest Works of the Metropolitan Sanitary District of Greater Chicago which treats about two tons a day of sewage sludge (dry basis). The largest wet air oxidation plant will be that for Borregaard Paper Co., of Norway. It should be in operation later this year or early next year. Purpose of the plant is to furnish 236,000 lb./hr. of process steam for plant use at 120-150 p.s.i.g. and so reduce imports of fuel oil.

ASPRO CLAIM BIG SAVINGS FOR NEW HANDLING SYSTEM

LESS costly raw material, quicker unloading and fewer process workers are the advantages given by Aspro-Nicholas Ltd., for the Tote system of bulk materials handling which they have adopted at their new Slough factory. This system, described in *CHEMICAL AGE*, 18 May 1957, p. 843, is supplied by Pressoturn Ltd., Leam Terrace, Leamington Spa.

It is used at Slough for the bulk handling of material received from Monsanto Chemicals Ltd., Ruabon, and starch from James Laing and Sons Ltd., Trafford Park. Weekly intake of material from Ruabon is about 10 tons of acetylsalicylic acid and from Trafford Park some 2-3 tons. The Tote bins, carried in both cases by the suppliers' own road transport, are unloaded with fork-lift truck. When needed for processing, the bins are transported from storage by fork truck to a screw-type tilt unit. This is elevated to the discharge position, the bin door opened and the material released into the elevating system for incorporation into the product.

After emptying, the bin is returned to store for checking, sealing and return to supplier. The Tote equipment at Slough comprises two tilt units and 50 74 cu. ft. capacity bins, each of which can take about 2,400 lb. of chemicals or 25 cwt. of starch.

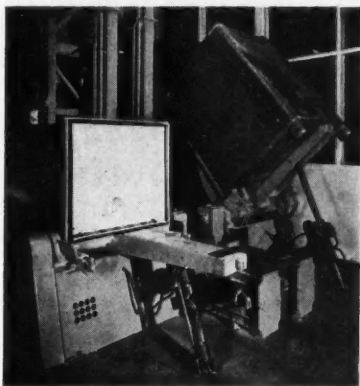
Aspro-Nicholas say that the reduction in raw material costs is due to savings made by suppliers. The system has eliminated non-returnable 10 lb. bags, 12 of which were packed in a returnable wooden case. One Tote bin replaces about 240 bags and 20 cases. The more compact bin increases the amount of raw material per delivery, cutting transport costs by about a third.

Advantages Claimed

The Aspro stores section has found that unloading is quicker and that the bin takes up less space per 100 lb. of raw material than the previous palletised load. Special lugs are said to give stability and easier stacking, while the cost of pallets has been saved. Handling in store is cut and there is no possibility of contamination.

Fewer process workers are needed to handle the bins as compared with the 10 lb. bags; the bins are unloaded automatically. Material losses have been almost eliminated, say Aspro-Nicholas, and documentation has been reduced. Dust, spillages and other hazards have also been reduced.

The bins, suitable for powdered, granular or liquid materials, are made in heavy gauge aluminium alloy. Filling aperture is 9 in. diameter and the discharge door 34 in. by 14½ in. Special gaskets and fasteners are said to ensure dust and weathertight sealing.



The two principal components of the Tote system: the Tote tilt with screw feed which controls supply of material into the Aspro process and a bin, which is seen tilted to the feed angle of 47°

Other items in the system, the Tote spinner head unit and the Tote jolter, are designed to ensure full capacity loading where required of fine powders. The Tote tilt discharger completes the equipment.

1954 Census Shows £67 million Rise in Gross Output of Chemicals

REPORT of the 1954 census of production on the general chemicals industry, published last week (HM Stationery Office, 2s.) shows that gross output in that year was valued at £333 million, against £266 million in 1951 and £164.1 million in 1948. Net output in 1954 was worth £134 million (£98 million in 1951, £65.2 million in 1948).

Capital expenditure in 1954, less amounts received for plant, machinery and vehicles disposed of, amounted to £34.1 million (£30.3 million in 1951, £12.3 million in 1948). Total labour force, including working proprietors, in 1954 was 91,000 (89,000 in 1951 and 86,600 in 1948).

Of the returns for the 1954 census, 456 came from establishments employing more than 10 persons; the gross output was valued at £323,660,000 (£255,194,000 in 1951). Average number of employees for these companies was: operatives 62,732 (62,904 in 1951); others 26,375 (24,365 in 1951). Net output per person employed was £1,473,000 in 1954 (£1,102,000 in 1951).

The following table is extracted from the census and shows sales of the principal products of the industry by larger establishments,

		1951	1954
Acids:			
Hydrochloric (1.14 spec. gravity) ...	Th. tons	236.9	256.0
Sulphuric (100% acid) ...	"	1,055.1	1,258.2
Alcohol: higher fatty	Th. cwt.	7.6	58.6
Sulphonated	"	52.0	76.7

Crosfields Install Automation In New £1 Million Plant

THE foundation stone of a new chemicals building for Joseph Crosfield and Sons Ltd., first stage in the £5 million rebuilding programme announced last year, was laid on 10 September by Mr. R. E. Huffam, a director of Unilever.

The new building is scheduled to be in full operation by July next year, with initial emphasis on the manufacture of alkaline detergents and silica gel. The somewhat traditional red brick exterior will house every relevant technical aid to production at present available to industry, and extensive automatic process control is being installed.

Workers' welfare is being fully provided for, and each plant employee will have twin lockers—one for working overalls and safety kit, the other for personal clothes, with drying facilities. Showers are being installed, and dining rooms provided for both day and shift workers, the latter having access to personal refrigerated compartments in which to keep food.

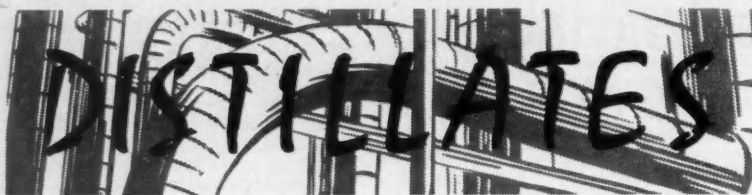
Chemical Production Index Up

The Board of Trade index of industrial production for May 1958 (based on average 1948 = 100) gives a figure for 'chemicals and allied trades' of 121, compared with 116 in April and 133 in May 1957.

Ammoniacal liquor (100% ammonia) ...	Th. tons	503.4	153.0
Bleaching materials ...	£'000	5,707	6,021
Calcium carbide ...	Th. tons	99.3	110.0
Carbons, decolorising and activated ...	Th. cwt.	222.3	115.2
Copper sulphate ...	Th. tons	48.5	48.6
Gases:			
Acetylene, dissolved	M. cu. ft.	443.6	502.1
Carbon dioxide ...	Th. tons	51.4	52.7
Hydrogen ...	M. cu. ft.	799.1	859.1
Nitrous oxide ...	M. gal.	142.8	173.4
Oxygen ...	M. cu. ft.	4,361.9	5,665.2
Butane, isobutane and propane ...	Th. tons	103.9	241.4
Nitrogen and others	£'000	5,186	10,843
Plasticisers; Phthalates	Th. tons	8.2	17.9
Phosphates ...	"	5.4	10.1
Other ...	"	3.3	2.1
Potassium cpds. ...	Th. cwt.	365.5	308.1
Rubber: Accelerators (nes) ...	Th. tons	5.0	8.5
Antioxidants (nes) ...	"	4.5	5.4
Sodium sulphate (all grades) ...	"	146.1	160.9
Sodium cpds., other ...	"	2,269.8	2,279.5
Titanium dioxide ...	Th. cwt.	716.8	1,270.8
Zinc oxide and sulphate	Th. tons	53.7	47.0
Metallic organic cpds. (not medicinal) ...	Th. cwt.	177.5	198.2
Sulphur ...	"	481.7	779.1

Saltney Firm May Not Extend, says Ministry

The Saltney fertiliser manufacturers, Edward Webb and Sons (Tower Bridge) Ltd., have lost their appeal to the Minister of Housing and Local Government for permission to extend their offices and laboratory and provide a canteen and other facilities, because the building would prevent the future widening of an unadapted road serving their factory and several others in the area.



★ **ONCE AGAIN** British scientists have shown the world that working in relatively small teams and to a 'shoe-string' budget they can produce results every bit comparable to those achieved by US and Soviet scientists who are backed by massive financial aid. The success of the Black Knight ballistic rocket at its first launching is a scientific achievement of the first magnitude, particularly as the cost to date, including research and development, production of the first rocket and those now in hand, is only between £4 and £5 million.

Mr. D. Lyons, head of the ballistic missiles division, Royal Aircraft Establishment, says that the Black Knight might reach a height of 600 miles and ultimately 1,500 miles as a two- or three-stage vehicle. As it is, at its first test the one-stage rocket reached 300 miles with a liquid fuel propellant. The second and third stages will use a solid fuel propellant.

I was delighted to hear from Mr. Lyons that a three-stage rocket could put up a 'worthwhile satellite'. The Royal Society, I learn, is to discuss the economics of a UK satellite project.

★ **WHERE** did ICI's simple but distinctive roundel originate? Older readers of this column who had dealings with Nobel Industries Ltd. will probably need no reminder, for this trade mark, now well known throughout the world, was formerly the trade mark of one of the four-founder companies of ICI.

It was in 1939 that ICI decided to adopt as their house mark one having similar features to the roundel of Nobel Industries. In it the name of the company was inscribed round the circle with the word Nobel over two bold wavy lines in the middle.

The mark was originally conceived by Mr. Ian Innes who sketched out the design while working in the engineering department of Nobel Industries. He is now in the work study section of Nobel Division's engineering department.

★ **PUBLICATION** last week of the 1954 census of production figures for the UK general chemicals industry underlines the fact that of all the industrial powers, Britain must be one of the most poorly served with statistics. Not only are the census results now nearly four years out of date, but they are also incomplete in too many details.

Production of individual chemicals is not given, results are shown as sales. The ideal, of course, would be to show rated capacities and actual outputs. Doubtless, the market survey sections of many of the larger chemical companies have more up-to-date figures based on intelli-

gent estimates and published information.

Whitehall lags far behind Washington in the compilation of statistics. To take one example, the US Bureau of Mines publishes far more comprehensive statistics relating to a wide range of materials, usually six months after each year end. Where actual results are not known, it is customary to publish official estimates.

In the 1954 chemicals census, far too many results are omitted or merged with others to avoid disclosing 'information relating to an individual undertaking'. This can be justified in only a very few cases. At present the practice is so widespread as to make nonsense of large sections of the census. Not only must the Board of Trade publish production statistics much earlier—this should not be difficult in this age of electronics—but they must also be much more comprehensive.

★ **WITH** THE centenary on 11 September of Hamilton Young Castner, it is worth recording that in England Castner found both the finance and engineering skill to develop the foundations of the electrochemical industry. This fact, believes Dr. D. W. F. Hardie, author of the *CHEMICAL AGE* feature 'Chemical Pioneers', should go a long way to explode the 'Britain-missed-the-chemical-bus' myth which arose chiefly in relation to lack of UK interest in dyestuff manufacture.

Born in New York on the eve of the American Civil War, Castner studied chemistry at the Columbia School of Mines under Charles F. Chandler. It was Chandler's idea that Castner, after several years as a consultant chemist in New York, should invent a process of sodium manufacture which would make its use commercially feasible for large-scale production of aluminium. Castner succeeded and for three years the Aluminium Co., Oldbury, of which he became managing director, produced sodium on a tonnage scale at less than one-fiftieth of its former cost.

Supercession of the Castner-Devil by the Hall and Heroult electrolytic processes for aluminium deprived metallic sodium of its only industrial application. Castner then brought into being a closely-knit pattern of inventions, each characterised by a combination of simplicity and novelty. This group of processes which Castner originated in the last ten years of his brief life were the beginnings of many of the modern developments in the manufacture of heavy chemicals from salt.

His principal inventions were: thermochemical sodium process (1886); electrolytic sodium process (1890); flowing mercury cathode caustic-chlorine cell (1892-3); sodium peroxide process (1890);

graphite anodes and graphitising process (1893); the first commercial solid non-chlorine bleaching agent; and sodium cyanide process (1894).

If lack of interest in dyestuffs put us at a disadvantage in 1914-18, Castner's cell equipped Britain to reply with interest when in 1915 chlorine was used as a war gas!

★ **US COMPETITION** to establish petrochemical units in Australia seems to be fairly intense. First in the field are the B.F. Goodrich Co., who in conjunction with Ampol Petroleum, are to set up a rubber factory costing £A3,350,000 in an outer suburb of Melbourne, Victoria. The site is near the new £A11 million factory being built by Fords of Australia. Key technical men from Goodrich are expected in Australia early in October.

Of the initial capital investment of £A3,350,000 Goodrich will hold 56.1 per cent, the remainder being held by Australian investors with Ampol having a major interest.

The American Vacuum Oil Co. are also interested in the establishment of petrochemical plants in Australia and last week, acting president, Mr. J. D. Rogers, said his company had been working on plants for some six months. He had just completed talks with Sir Thomas Playford, Prime Minister of South Australia, on the construction and working of plants in both South Australia and Victoria.

★ **THE IMPORTANCE** of fluorine, from which is now developing a new field of chemistry, has been recognised for some time and this knowledge in fact prompted *CHEMICAL AGE* to report exclusively two recent symposia on the subject. Further new developments in the chemistry of fluorine were forecast last week by Mr. H. R. Leech, assistant research manager for ICI at Widnes when he said that through its use, new plastics with remarkable properties were becoming available.

Fluorine is being used in the production of new non-inflammable anaesthetics and considerable quantities are called for in atomic energy, and in refrigeration. The production of fluorine has been reduced to a simple routine and Mr. Leech felt that the industrial development of the material itself had hardly begun. New kinds of compounds are constantly being made available and their potential value explored.

Birmingham University, where much fundamental work on fluorines is being carried out, will in fact be the venue next year for an international symposium on fluorine chemistry.

Alembic

Birmingham Microchemistry Symposium-3

Useful Methods in Functional Group Determination of Organic Substances

METHODS of identification of an unknown substance by reorganising its different functional groups, by preparing derivatives of some or all of these groups and by determining the equivalent weight with respect to such groups were surveyed by Professor Stig Veibel (Department of Organic Chemistry, University of Technology, Copenhagen). In particular, his remarks dealt with the identification of such groups which do not themselves provide the possibility of simple titration.

Hydroxyl group: A very accurate method for determining the hydroxyl group is by conversion of many primary and secondary alcohols into xanthates. From the equivalent weight of the xanthate that of the alcohol is found by subtracting 114.2. Tertiary alcohols which do not form xanthates might be identified as tertiary alkylthiuronium picrates. In this case the equivalent weight of the alcohol is found by subtracting 288.2 from the equivalent weight of the picrate. This method fails however if only one of the radicals is an aryl or aralkyl.

More general is a reaction between *m*-nitrophenyl-*iso*-cyanate and hydroxyl compounds, resulting in the formation of *m*-nitrophenylcarbamate esters. The equivalent weight of the ester can be estimated titrimetrically, and the equivalent weight of the hydroxy compound is then found by subtraction of 164.1.

Heterocyclic Compounds

α or β -Hydroxy acids or esters may cause difficulties as they can be converted into heterocyclic compounds by ring closure, giving off water or an alcohol, so that all esters of such acids will give the same derivative as the acid itself.

Aromatic hydroxycompounds are conveniently estimated through the aryloxyacetate acids formed by reaction, between the sodium salt of the phenol and sodium chloroacetate. The acid is liberated, recrystallised and titrated with standard NaOH. By subtracting 58.0 the equivalent weight of the phenol can be found.

Sulphide: Aliphatic sulphides can be characterised and estimated by converting them into dialkyl *p*-bromophenacyl sulphonium picrates. The test is valid for alkyl sulphides only but not if the sulphide group is very mobile.

Carbonyl compounds: These are conveniently isolated as derivatives of a substituted hydrazine, such as semicarbazide, *p*-carboxyphenylhydrazine or 2,4-dinitrophenyl hydrazine. For the equivalent weight of the semicarbazone that of the carbonyl compounds is calculated by subtracting 57.1. With *p*-carboxyphenyl-

This summary of the paper by Professor Veibel begins our third report of the International Microchemistry Symposium. Others appeared in our issues of 30 August and 6 September. Full proceedings will be published by Pergamon Press.

hydrazones, the equivalent weight of the carbonyl is found by subtracting 134.1 from that of the *p*-carboxyphenyl hydrazone. The formation of heterocyclic compounds from different types of diketones, ketoacids, hydroxyketones or unsaturated ketones and hydrazine derivatives have to be borne in mind when evaluating the equivalent weights found.

Use of 2,4-dinitrophenyl hydrazones in a micromethod for the estimation of carbonyl compounds had been suggested. A condition for the usefulness of this method is that the carbonyl compound is quantitatively converted into the 2,4-dinitrophenylhydrazone and that this substance is so insoluble in the solvent used that it does not interfere in the estimation of the excess of the reagent. This condition was not always fulfilled, reported Professor Veibel. The same difficulty arises in the estimation of carbonyl compounds by reducing them with sodium boron hydride. Substituents at the carbonyl carbon atom diminishing

the polarisation of $C=O$ -double bond will counteract the reduction of the carbonyl compound, thus invalidating the estimation.

Carboxylic acids: While titration with standard NaOH is the most usual method of estimating these acids for determination of the acid contained in an ester, it is often convenient to isolate the acid as a derivative, eg, as a salt, and then to determine the equivalent weight of the salt. Veibel said that he had found it convenient to isolate the acid as the benzylthiuronium salt. Its equivalent weight was determined by titrating a solution in glacial acetic acid with perchloric acid and subtracting 166.2 + *n*.18 from the equivalent weight found. (The *n*.18 was added as the salts often crystallised with water of crystallisation.)

Radiochemical methods: With regard to the various methods recommended for determination of functional groups, Professor Veibel considered that those making use of radioactive isotopes would prove of most value as microchemical methods. Three different types of estimation had been recommended: the isotope-dilution method; the reversed isotope-dilution method; and the derivative method.

The first method can be used for determination of the amount of an unknown substance present in the mixture. The second method is used for the estimation of the amount of an unknown radioactive substance present in a mixture, while the derivative method depends on the quantitative reaction of an unknown substance (X) with a radioactive reagent (Z) and the quantitative isolation of the derivative XZ. This method could be very well adapted to chromatographic estimations, Professor Veibel remarked.

Developments in Methods of Determining Alkoxy

RECENT developments in the methods for determining alkoxy were discussed by Dr. W. I. Stephen (Department of Chemistry, Birmingham University), under the separate headings of apparatus, wash liquids, absorbing solutions and alternative procedures. There had been, in particular, significant developments over the last five years from Swedish laboratories due to their interest in cellulose.

A procedure for determination of alkoxy groups was first suggested by Zeisel in 1885. This had been followed by the Pregl apparatus and in 1954, the alkoxy apparatus published in British Standard 1428 Part CI, became the standard apparatus. It had been developed by Belcher *et al.* and could be used for micro determinations.

The next method was that using Dr. W. Kirsten's apparatus, where digestion is carried out in a separate vessel. The Swedish chemist G. Gran had published

details of another apparatus which Dr. Stephen said was of value in industrial work. At Birmingham a fairly exhaustive investigation of sub-micro determination (about 50 μ g.) was being carried out, and Beckett, Belcher and West had now published some of their work: For vanillin 50 μ g. containing 20 to 21 per cent alkoxy, 200 μ l 0.01 N $Na_2S_2O_8$ was used. It had been possible to analyse a very wide range of samples with a standard deviation of ± 0.22 to 0.25 per cent.

Use of sodium thiosulphate showed that aqueous solutions react with methyl iodide and therefore it had been suggested that it should not be used for scrubbing methyl iodide. At Birmingham, there was a preference for sodium antimonyl tartrate which had been found effective as a wash solution. If sulphur was not present in the sample water was as good as any other washing agent.

In the cellulose industry there was a

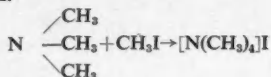
preference for antimonyl sodium tartrate or cadmium sulphate. Some ICI workers had suggested sodium acetate while other wash liquors were red phosphorus, 5 per cent sodium bicarbonate, and red phosphorus in 5 per cent cadmium sulphate solution. Water and cadmium sulphate were used by some workers to remove sulphur-containing compounds, while Kirsten used sodium bicarbonate. Dr. Stephen said, however, that 1 per cent acetic acid served equally well.

If sulphur were present, alkaline wash liquids should be avoided, since methyl iodide could react with the sulphur to form mercaptans and hence some methyl iodide was lost. There were advantages in using 5 per cent cadmium sulphate or 10 per cent antimonyl sodium tartrate.

There was general agreement that thio-sulphate wash liquids were detrimental, Dr. Stephen reported. An alternative to the classical absorbing solution had been

found in Gran's modification of the Willstätter and Utzinger procedure, which was selective for methoxyl, but also permitted simultaneous determination of methyl and ethoxyl.

In Gran's modification triethylamine is used.



and the product formed is found to be appreciably soluble in ethanol. In isopropanol solubility of the salt is much less. Use has been made of this finding for distinguishing methoxyl groups from ethoxyl groups. Gran claims an accuracy of ± 2 per cent and that the estimation can be carried out in one hour.

(N.B.—Time did not allow Dr. Stephen to discuss a colorimetric method and submicro determination, as had been expected.)

Problems of Alkoxy Determinations Outlined in Discussion Period

Dr. F. Feigl corrected a statement by Prof. Veibel about the use of spot tests for isomeric substances. It was not true that spot tests could substitute for classical methods. One reaction he would mention in particular, namely, $\text{ROH} + \text{CS}_2 + \text{KOH} \rightarrow \text{ROC S}_2 \text{ K}$, indicated by Prof. Veibel. This reaction, said Professor Feigl, could not be translated into chemical test reaction but one could carry out the following $\text{OCH}_3\text{HI} \rightarrow \text{CH}_3\text{I} \xrightarrow{\text{BAC/Br H}_2\text{O}} \text{I Br} \xrightarrow{\text{K I}} \text{IO}_3 \rightarrow 3\text{I}_2$. However, $\text{KI} + \text{H}_2\text{C}_2\text{O}_4$ has to be substituted for HI. Spot tests were excellent for identification but not for determination. Thirty years ago he had been criticised for spot test analyses. The new ways in qualitative analysis were only six years old but no doubt in due time they would find their place and acceptances.

Alkoxy and Alkylamine Determinations

Dr. Steyermark (Hoffmann-la Roche, US) wanted to call attention to carrying out alkoxy and alkylamine determinations. Although one could carry out alkoxy determinations with alkylamines, by first boiling with HI, followed by dry distillation, the reasoning was faulty, however. He had seen the liberation of MeI in boiling HI where no alkoxy was present. He gave a warning about mistaking N-Me for O-Eth or O-Me.

Dr. W. Kirsten indicated he had thought about these problems. He had wondered about freezing out products and using gas chromatography to get a complete spectrum of the elements present. Dr. Veibel had mentioned detection of carbonyl groups by isolating these as derivatives of a substituted hydrazine and he had said that there were a few instances when this test did not work. Dr. Kirsten had also tried this method and found that it didn't work at all. Seldom was the precipitate insoluble and a much stronger colour developed than that

of phenylhydrazine. 3-phenyl trinitrohydrazine could be shaken out with carbon tetrachloride. The excess of 3-phenyl trinitrohydrazine could be reacted with dimethyl hydrobenzyl. The product obtained could be dissolved in dimethylformanilide and a violet colour was obtained which could be measured.

Dr. Kirsten took the opportunity to correct a statement by Dr. W. I. Stephen, who had stated that, using the Kirsten apparatus for alkoxy determinations, the tube and contents were heated in a steam bath. In actual fact, they heated the tubes in a water bath.

Dr. A. Lacourt (Universite Libre de Bruxelles, Belgium) inquired about the rate of gas flow through the apparatus used by Dr. Stephen at Birmingham.

Dr. Stephen replied that the gas flow was regulated to two bubbles per second during distillation.

Dr. Kirsten wanted to know if anyone had been completely successful in determining alkoxy compounds containing sulphur. He had tried but had not had any success.

Good Results with Simple Bubbler

Mr. G. Ingram said he had been analysing xanthates and had had no trouble using a very simple apparatus—it was a simple bubbler in fact, and the gas flow was varied. Knowing the purity of the xanthates, he had found no trouble at all, and results were good.

Dr. Kirsten wished to know whether sulphide was precipitated.

Mr. Ingram remarked that it had no effect. The error with sulphur compounds was not due to sulphur, considered Dr. Kirsten. He had obtained no results with compounds which form sulphides.

Dr. Steyermark informed Dr. Kirsten that with regard to sulphur analysis in compounds containing sulphur, using an AOC apparatus and NaAc solution, HI

was almost dark brown or black. He purified by refluxing in CO_2 and N.

Dr. Kirsten agreed with this. Ordinary compounds with fixed sulphur caused no trouble. But if sulphate was present in boiling solution then NaAc would give no results. With large amounts of sulphate there were low results with acetate or sodium bicarbonate.

Mr. A. F. Williams (ICI Nobel Division, Ardeer) referred to Dr. Kirsten's suggestion of gas chromatography. At the analytical conference at Lisbon, the use of gas chromatography for determination of iodine had been mentioned in a paper by Martin.

Mr. C. Tomlinson (Liverpool) commented on the wash solution of 5 per cent cadmium sulphate and 5 per cent thio-sulphate used at Liverpool. The gas flow was 15 ml./min. Methyl iodide had considerable solubility in water, he said. Was Dr. Stephen sure that the methyl iodide would react with the sodium sulphate used as wash solution? Dr. Stephen referred Mr. Tomlinson to Ingold's paper, and in 1956 Savill (*Chemistry and Industry*) reported the use of methyl iodide with sodium thiosulphate as a means of detecting sulphides. Dr. Stephen said, however, that he saw no reason for using sodium thio-sulphate if other wash solutions had proved reliable.

Reaction Can be Avoided to Some Extent

Dr. Kirsten stated that there could not be the slightest doubt that sodium thiosulphate reacts with methyl iodide, but he thought reaction could be avoided to some extent. He had had thick white precipitates of sulphur and very low results using thio-sulphate. If no hydrogen sulphide was formed, water could be used, although methyl iodide was somewhat soluble in water.

Mr. F. J. Armson (G.K.N. Group Research Laboratories, Wolverhampton), said that water as the wash liquor was satisfactory, except for an amendment. Above 0.5 in a hundred it was possible but if water was at 42°C better results were obtainable; but with regard to the use of sodium thiosulphate, he accepted Dr. Steyermark's 100 per cent recovery. Having an electrolyte present, he believed, decreased the solubility of methyl iodide.

Mr. Ingram said he had found water satisfactory, but there was a dependence on the state of HI and on the type of compound being analysed. If the compound decomposed carbon charring might occur, and some iodine could be taken over.

Dr. Stephen pointed out that if absolutely accurate results were required fuller precautions had to be taken in washing. If carbon dioxide was used as the gas stream the carbon dioxide suppressed the solubility of methyl iodide in water.

Mr. Tomlinson asked Dr. Stephen if he had any experience with solvents such as pyridine.

Dr. Stephen said no, only with phenylpropionic anhydride.

Mr. B. Seaton (Plessey, Northants) asked whether solvents should be used at all. Also he wondered about the use of mixed solvents. Was there any leaking at the joints in the British Standard apparatus?

Dr. Stephen said they had no leaking at the joints.

Mr. Ingram said he had not used any solvent for 12 years.

Dr. Kirsten had found the opposite. His laboratory had carried out for a long time analysis for a wood research unit. This unit then stated that his (Kirsten's) analyses were all wrong. At that time he was using pyridine. He had therefore obtained pure compounds, but his results were still poor. Using phenol and propionic acid, his results had been much better, but in a few cases his results had still been very poor. He had thought that reaction time might be involved. He had therefore heated the compounds in steel tubes in a water bath. Only two compounds didn't give good results. After heating for one and a half

hours, better results were obtained. With the isopropoxy compounds, no good results were obtained due to steric hindrance of the iodide.

Dr. A. F. Colson (ICI, Alkali Division, Cheshire) asked Dr. Stephen if the BS apparatus was effective with very volatile materials? Dr. Stephen thought that if a capillary was used, one might get away with it but in that case, use of Kirsten's or Ingold's apparatus was recommended.

Dr. Colson said that he placed in the scrubber above the reaction vessel glass beads wetted with H₂O and good results were obtained.

Dr. Stephen informed Dr. Colson the BS apparatus was not very suitable for highly volatile materials.

Modified Pregl-Dumas Method for Microdetermination of Nitrogen

FAILURE of the original Pregl-Dumas method for the micro-determination of nitrogen to give accurate results with certain types of compound including some purines, pyrimidines, polymers, long chain fatty acid amides, mineral oils, and compounds containing the pteridin ring structure, was usually attributed to incomplete oxidation of methane produced during decomposition of the sample, or to retention of nitrogen by carbonaceous residue derived from the sample.

Dr. A. F. Colson (Alkali Division, Imperial Chemical Industries Ltd., Norwich) outlined in his lecture the many attempts which have been made to improve the Pregl-Dumas method and some of the modifications that had been described.

In addition to the methods in which oxidation of the sample was promoted by the introduction of oxygen, the several procedures that were based upon the use of temperatures much higher than those used in the normal Pregl-Dumas method were described.

In the final form of Kirsten's method, (*Analyt. Chem.*, 1957, **29**, 1084) nickel oxide at 1,000° to 1,050°C was used as the oxidising reagent in the combustion tube and the sample was heated alone in a boat at 1,050 to 1,100°C. Hopcalite at 150°C was used to recombine any dissociation products of carbon dioxide. The method was not subject to errors due to the retention of nitrogen by carbonaceous residue from the sample since at the high temperature used any carbon in the residue was converted to carbon monoxide.

Modifications had been made to Kirsten's method by Parks and Belcher, and Dr. Colson had also devised a modified procedure. In this the permanent filling of copper oxide and copper, and the sample mixed with copper oxide, were heated at 900°C, and any oxygen or carbon monoxide was removed by passage of the gaseous combustion products over copper oxide and copper at 600°C. Details of the method were given together with some experimental evidence in support of the view that the macro-Dumas method was more subject to errors due

to the incomplete combustion of methane than is the normal Pregl-Dumas micro-procedure and that the latter was probably less liable to error from this source than was suggested in the literature.

In Colson's modification of the Pregl-Dumas method which was developed before the introduction of the high-temperature methods, an attempt was made to devise an alternative procedure involving the use of copper oxide and metallic copper only. As a preliminary, the incomplete combustion of methane in the normal Pregl-Dumas method was confirmed by some simple experiments. From the results obtained in these experiments it was concluded that if in the normal Pregl-Dumas procedure a small amount (e.g. 1 ml.) of methane was produced about 96 per cent of it would be completely oxidised. Therefore the possibility of using higher temperatures to achieve complete oxidation of methane had been examined.

From a series of blank experiments, it was found (by visual inspection) that at temperatures between 750°C and 1,000°C, the micro-bubbles rising in the nitrometer were larger than those obtained at 720°C and showed no decrease in size even after prolonged passage of carbon dioxide through the system. This was thought to be due to dissociation of carbon dioxide but it was later established by further experiments that the main cause was the liberation of oxygen from the hot copper oxide.

The method was applied to the analysis of a number of compounds including *m*-dinitro-benzene, 1-chloro-2:4-dinitro-benzene, and *p*-nitroaniline, for which the normal Pregl-Dumas procedure was known to be suitable and it was found that the two procedures gave results of equal accuracy. In these analyses the sample was mixed with fine copper oxide and was heated to about 900°C.

Application of a modified method to analysis of compounds which give low results by the normal Pregl-Dumas method due to retention of nitrogen has not been extensively investigated, but the method had been found to give satisfactory results for such substances as cer-

tain polymeric compounds. Dr. Colson stated, however, that his modified method was not applicable to all types of compound for which the normal method was unsuitable. It failed with some very complex cyclic compounds. It was believed that the most satisfactory procedure would be some method involving the use of gaseous oxygen.

Methane as a source of error in the Pregl-Dumas method was discussed by Dr. Colson. The normal macro- and micro- forms of the Dumas method for determination of nitrogen were known to be subject to errors due to incomplete combustion of methane. In the micro procedure this effect had been observed when relatively large weights of sample had been used as in the analysis of universal oils. But there was reason to believe that this source of error was more pronounced in the macro-method than in the micro-procedure. Some support for this had been provided by experiments by Haas (*J. Chem. Soc.*, 1906, **81**, 570).

In analysis of compounds containing two or more methyl groups attached to carbon, more accurate results were obtained by the normal Pregl-Dumas method that had been expected and could be accounted for only on the assumption that the methane produced by the combustion of the sample had been completely oxidised. Dr. Colson suggested that a possible explanation of the incomplete oxidation of methane in the macro-Dumas procedure was that the surface area of hot copper oxide presented to the gaseous combustion products of the sample was less than in the micro method. Approximate calculation indicated that whereas contact times were the same in each case, the ratio of weight of sample used for the macro-determination, to that taken for the micro-determination, was about 20:1 and the ratio of corresponding surface areas was only about 4.1 per cent.

Discussion

Mr. G. Ingram (Courtaulds Ltd., Berks), who chaired this meeting, thanked Dr. Colson for his paper, and said he was intrigued to hear about Dr. Colson's remark on oxygen.

Dr. Al Steyermark (Microchemical Department, Hoffmann-la Roche Inc., New Jersey, US), stated that there was an error which occurred in Dumas estimations, but it was not due to methane. Apparently, methylamine formed especially when there were many methyl groups. Ammonia could be got rid of, but methylamines could still be detected. He believed methane went through the Dumas train and was absorbed in the caustic.

Dr. W. J. Kirsten (Uppsala Universitets, Medicinisk-Kemiska Institution, Uppsala, Sweden), suggested that Dr. Steyermark extended his work and sent out various samples for examination.

Dr. Steyermark said he would be doing this. With regard to the N-N or O-N linkage, phenylhydrazine could be taken and a perfectly good compound was obtained after reduction, as long as the N-N or O-N were not in a ring. Dr. Steyermark said that an article would be appearing in the September *Analyst*.

ADVANCES IN ORGANIC ANALYSIS DUE TO ULTRA-MICRO METHODS

THOSE older classified micro-methods that have been found to stand the test of time in an organic analytical laboratory were reviewed briefly by Dr. John Haslam (Plastics Division, Imperial Chemical Industries Ltd., Welwyn Garden City). He then made some pertinent observations on other forms of organic analysis. There were great possibilities inherent in the new field of ultra-micro analysis, he thought.

The position with regard to the ultra-micro balances available was very satisfactory, and Dr. Haslam had no doubts that amounts of sample of the order of 50 μ g. could readily be weighed to within ± 0.03 μ g. on a modern prototype quartz fibre balance with the following provisos: (1) the load on each pan should not be greater than 250 mg., and (2) weights on the individual pans should not differ by more than 1 mg.

Dr. Haslam considered that Unterzacher (*Microchim Acta*, 1957, 448) was well on the way to bringing determination of carbon and hydrogen in organic compounds on to a volumetric basis by his process. There were difficulties to be solved and, particularly with liquids, there were handling difficulties.

Providing there were no sampling difficulties, there was a tendency towards use of semi-micro methods of organic analysis, and Dr. Haslam referred to two with which he had been connected.

N in Synthetic Rubbers

In determination of nitrogen in synthetic rubbers, he had found that conventional Kjeldahl procedures using sulphuric acid, selenium and sodium sulphate followed by distillation of the ammonia in ammonium compounds tended to give low results. He used the simple principle developed by E. R. Ling many years ago for the rapid determination of nitrogen in milk. The method was speedy and troublesome distillation of ammonia was avoided. The amount of hypobromite used up in the test was determined by reaction with iodide and acid, the liberated iodine being titrated with standard sodium thiosulphate solution.

At the plastics division, his laboratory had been called upon to develop a very rapid method for the semi-micro determination of chlorine in small samples of polyvinyl chloride (p.v.c.) and related polymers, e.g., copolymers of vinyl chloride with vinylidene chloride or vinyl acetate. The method had to be applicable to larger numbers of samples of the order of 20 mg. each at one time. A special bomb had therefore been designed (2 ml. capacity) in which 20 mg. of the polymer could be fused with 1.0 gm. of sodium peroxide and 0.06 gm. dry starch.

The reaction product was extracted with water and the extract made faintly acid with nitric acid prior to titration, in a volume of 30 ml. with N/50 silver nitrate solution to a pre-set e.m.f. difference between a silver wire as indicator electrode and a silver wire in contact with a weak solution of silver ions as reference electrode. This latter titration was carried out automatically.

Analysts in organic industries, it was felt, might not have made as much use of electrolyte procedures as they might profitably have done. In plasticiser identification, the method indicated by Haslam *et al.* (*J. Appl. Chem.*, 1951, 1, 112) had been employed, but it was desired to detect methyl alcohol in aqueous solutions. Work by A. Zapletalek (*Coll. Czech. Chem. Comm.*, 1939, 11, 28) had suggested, however, the electrolytic oxidation of methyl alcohol to formaldehyde under controlled conditions. The methyl alcohol solution was made 2N in sulphuric acid, and a current of 15 to 20 mA passed for a period of 30 minutes maintaining the solution at 60°C. A circular platinised platinum electrode was the cathode and a circular piece of platinum foil with holes punched in it the anode.

Positive Results

The chromotropic acid test was applied to the resulting solution for formaldehyde. Positive results were obtained readily on 0.5 per cent V/V methyl alcohol solutions even in the presence of large proportions of ethyl alcohol, which did not respond to the test. The validity of Zapletalek's test was also confirmed.

Gas-liquid chromatography (G-LC) was then considered by Dr. Haslam. He had found the gas-liquid chromatographic test was useful in the direct comparison of monomer made by different processes. Moreover, using G-LC procedures it has been found possible to isolate pure products. On occasion, the principle of the test can be extended to examination of polymers. Thus small proportions of polyethyl esters could be detected and determined in polymethyl methacrylate.

Interesting features about the test were: the complete conversion of the alkoxyl groups in the polymers to the corresponding alkyl iodides; the purification and drying of these iodides and their quantitative collection in *n*-heptane; and determination of the amounts of the respective iodides by G-LC examination of the *n*-heptane solution of the alkyl iodides in the presence of two markers, methylene dichloride for methyl iodide and ethylidene dichloride for ethyl iodide. It was felt that the principles involved might be of interest to many organic chemists concerned with the

Zeisel test. Its application had already been extended to a case where it was suspected that a particular polymer contained a polymerised vinyl isobutyl ether. Pronounced evidence was given of the presence of isobutyl iodide in the products of reaction of the polymer with hydriodic acid.

Dr. Haslam hazarded the opinion that it was very probable that a considerable number of tests given in Feigl's book on *Spot Tests*, Vol. II, 'Organic Applications', could, with advantage, be placed on a quantitative basis. He had used Feigl's test (see p. 129, Feigl's book) to determine small amounts of alcohols (in the absence of esters) in hydrocarbon solvents. The method is based on the production from the alcohol of the corresponding alkali/alkyl xanthate by reaction with carbon disulphide and alkali. This alkali/alkyl xanthate is then reacted with molybdate in acid solution to yield a coloured chloroform soluble reaction product which is measured at 585 m μ .

Examining Polyamides

Attention was drawn to a method of examination of nylon and related polymers based on their hydrolysis with acid. The products of hydrolysis were evaporated to dryness, then dissolved in alcohol and the alcohol solution spotted on filter paper. Elution with a mixture of *n*-propanol, ammonia and water was then carried out for seven hours. After drying the papers, the various hydrochlorides and acids present in the hydrolysis products were detected by means of ninhydrin and methyl-red borate buffer sprays.

By means of high voltage electrophoresis, however, Dr. Gross was able in 10 minutes to separate quite clearly hexamethylene diamine dihydrochloride, ϵ -aminocaproic acid hydrochloride and diaminodicyclohexylamine dihydrochloride. Dr. Haslam considered that Dr. Gross's work would have considerable influence in the future.

Automatic titrimetry was finding increasing application in many organic laboratories; but attention should be paid to those titrations where the end point or end points were not *a priori* known. For this purpose, Dr. Haslam considered there was a need for a more simple form of burette, i.e., other than the mercury piston burette geared to the pH and recording system. For titrations in non-aqueous media, the procedure outlined by Cundiff and Markunas (*Analyt. Chem.*, 1956, 28, 792) and Fritz and Yamamura (*Ibid.*, 1957, 29, 1079) had been used successfully to determine quite small amounts of salicylic acid in phenyl salicylate even when appreciable amounts of phenol were present.

Reference was then made to P. B. Sweetson's Wickbold oxyhydrogen combustion method for decomposition of organic fluorine compounds for determination of fluorine. The method was rapid and eliminated the difficulty resulting from high salt content normally present after a Parr bomb fusion.

It was suggested that there were possibilities for the analyst in the principle

of zone melting and also in the ASTM infra-red index. Much valuable time could be saved by using the index. Dr. Haslam also recommended that more use should be made of the phenol-hypochlorite reaction for ammonia (*Analyst*, 1956, **81**, 64) for it often succeeded where other methods failed.

Dr. R. Taylor London gathered that Dr. Haslam's laboratory standardises on paraffin wax and tritoyl phosphates as the stationary phase. Had Dr. Haslam any evidence that tritoyl phosphate was stable at temperatures over 100°C?

Dr. Haslam said—Yes, he used the procedure, particularly with substances which boiled at 150°C. With adhesives, one did not need a higher boiling point. Infra-red spectroscopy had been mentioned by Dr. Haslam, another delegate remarked, as giving a good idea where a mixture of plasticisers was involved. He asked Dr. Haslam whether he had used column chromatography with such products, and whether he had had experience with other polymers other than methacrylate.

Dr. Haslam stated that he had had no direct success with polymers and he had not done well with two or three substances forming a mixture. He had not made much use of column chromatography. He started with polymethacrylate in vacuo and obtained first-class polymethacrylate. If the same process was carried out on p.v.c., too many breakdown products were obtained. Every man had to work out for himself all known compounds.

Mr. J. Fraser said that Dr. Haslam had mentioned positive differential in vacuo

with the gas-vapour phase chromatography. Had he had an experience with high boiling point solvents?

In reply, Dr. Haslam said that what tended to happen was that one started with these and continued with high pressure. They did recover solvents and examined these, using vapour-phase chromatography.

Mr. T. R. F. W. Fennel remarked that in the list of elements mentioned by Dr. Haslam, did he use micro methods with samples of 3 to 5 mg., and for semi-micro methods did he use a sample of 10 to 50 mg.? He asked Dr. Haslam's opinion on whether he preferred to use, for sulphur, phosphorus, chlorine and fluorine, a closed bomb or Carius method or the oxygen method?

Dr. Haslam informed Mr. Fennel that for semi-micro work he used 20 to 50 mg. and 4 to 5 mg. for new polymers. With sulphur, he used the Carius method and worked under pressure. For chlorine, he used a Carius method and a sealed tube. He did not carry out semi-micro tests very often.

Another member of the audience said that she had carried out some micro-estimations on carbon and hydrogen, which depended on combination of substances freezing out of carbon dioxide and water and subsequent volatilisation and measurement of the appropriate pressures.

This was extremely interesting information, thought Dr. Haslam. The method was likely to be a volumetric one. She had simply sealed down the method, of course. He, and others he believed, would be interested in following this work.

Chromatography of Larger Molecules, by Tudor Jones

PROBLEMS peculiar to the chromatography of the larger molecules as distinct from small molecules, arose mainly when the scale of separation was designed to provide material for further work, e.g., as intermediates for organic synthesis or for biological testing. This was stated by Tudor S. G. Jones in his lecture on chromatography of the larger molecules.

Elution development, where the scale had to be kept small, had been applied to many classes of organic compound of high molecular weight, it was reported. Both paper and column chromatography had been used, with solvents of constant, systematically varied and continuously varied composition, and using predominantly adsorptive and predominantly partition stationary phases. Ion exchange resins had provided the most versatile of the chromatographic media, and in both elution analysis and displacement development, the best separations had been achieved when material of lower cross-linking had been available.

For efficient chromatography of the larger molecules, it was necessary that the pore size, determined by the degree of cross-linkage, should be sufficiently large for the rapid diffusion of the substances to be chromatographed into and out of the resin particles. Low cross-linkage was available in the sulphonated polystyrene resins, and these had been used extensively

for peptides. Proteins had been separated mostly on polycarboxylic resins, of high cross-linkage, and although almost exclusively a surface phenomenon, capacity was remarkably high. Anion resins had been used for phosphoryl peptides and for polynucleotides.

Displacement development offered great advantages in the scale on which it could be carried out, Dr. Jones remarked. With low cross-linkage, the bulk of the resin could be saturated by the mixture to be separated before development by the displacing substance. High capacity was thus a feature of the displacement development method. It had been used for the separation of carbohydrates, peptides, fatty acids, penicillins and antihistaminic drugs. Carrier displacement, in which the substances to be separated were sandwiched between zones of related substances from which they might be easily isolated by classical means, overcame one of the main difficulties—that was the necessity for detecting the change from one zone to the next, contiguous zone. Fatty acids, interspersed by their esters, provided a good example of carrier displacement. Irreversible adsorption was one of the difficulties with large molecules for which a solution was sometimes very hard to find. Substances containing many phenyl groups and strongly basic compounds suffered from this disadvantage.

Microdetermination of Functional Groups, by Professor Ma

MICRO-DETERMINATION of functional groups was defined by Professor T. S. Ma (Brooklyn College of New York City, US) as the analytical methods for the quantitative determination of organic functional groups using approximately one-tenth milli-equivalent of the compound. This deviation from the conventional limitation of organic micro-analyses to the range of a few milligrams was based on the consideration of several factors: (a) 0.1 milli-equivalent of material consumed 10 ml. of 0.01N titrant; (b) 0.2 milli-mole of gas occupied about 2 ml. volume; (c) end-product weighing over 10 mg. could be handled on a semi-micro balance; (d) the sample or end-product was usually recoverable after the determination for functional group, and hence the problem of conserving the sample was not critical.

There were many more functional groups than the number of elements present in organic substances. It was pointed out by Professor Ma that the current laboratory manuals on quantitative organic micro-analysis cover only a few functional groups. A proposition was made to classify the various functions into five categories: (1) oxygen functions; (2) nitrogen functions; (3) sulphur functions; (4) unsaturated functions; and (5) miscellaneous functions.

The project of investigation which had been carried out during the past seven years by students at Brooklyn College and New York University was presented. It consisted of: (a) offering a course to teach functional group analysis in which each student analysed about 15 different functions according to prescribed directions; and (b) assigning research problems to thesis students to study the applicability and limitations of the various methods, as well as the possibility of improving the apparatus and procedure.

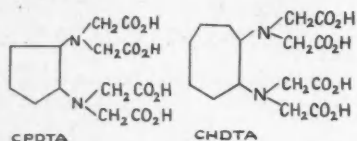
Chemical Reactions and Laboratory Equipment

Chemical reactions and laboratory equipment involved in the project were described. A number of functional groups could be determined on the micro-scale using the conical flask, 10-ml. burette and other general laboratory equipment. To this class belonged the basic functions, some acidic functions, hydronyl groups, 1-2 diols, phenols, esters, carbonyl groups, sulphhydryl, isocyanate, isothiocyanate groups, etc. Then there were apparatus which were specially designed to determine one or more functional groups, such as the apparatus for acyl, alkoxyl, alkimid groups, methyl side chain, very weak acids. Other apparatus were for micro-determination with the Karl Fischer reagent or titanous chloride, for gasometric determination of the amino, hydrazino groups, active hydrogen, unsaturated linkages, disulphides, etc. This project was near completion and the results would be published in book form.

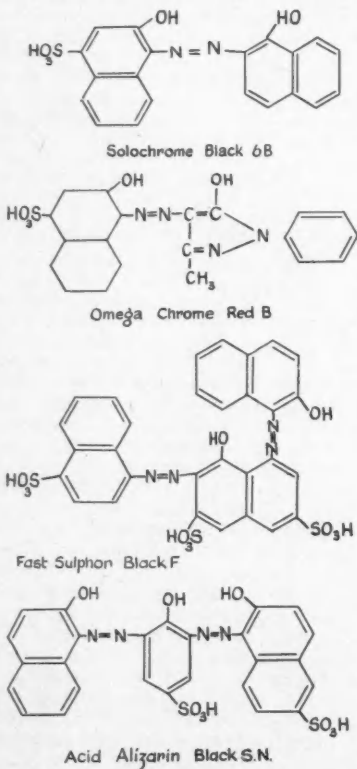
DR. WEST ON NEW REAGENTS IN COMPLEXOMETRIC ANALYSIS

IN his paper 'New Reagents in Complexometric Analysis', Dr. T. S. West (Chemistry Department, Birmingham University) discussed some recent work on the development of reagents for complexometric analysis which he and his co-workers had carried out in the school of analytical chemistry at Birmingham University. Two main approaches are possible: (a) the development of chelating agents which are more selective in their action than EDTA, (b) the evolution of better and more selective metal ion indicators.

Among the new chelating reagents, 1,2-diaminocyclopentane-N,N,N',N'-tetraacetic acid (CPDTA) and 1,2-diaminocycloheptane-N,N,N',N'-tetraacetic



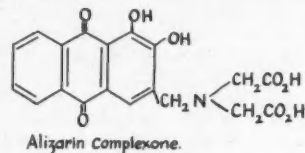
acid (CHDTA) are outstanding. Polarographic studies have shown that CPDTA is roughly equal to EDTA in chelating power, but is considerably slower in its action. CHDTA, on the other hand, is more powerful by about five orders of magnitude. Two simpler reagents, namely 1:2 diaminopropane, N,N,N',N'-tetraacetic acid and 2,3-diaminobutane tetraacetic acid are more powerful than EDTA in their action though less so than



CHDTA, but are more readily synthesised and react rapidly with most cations.

Solochrome Black 6B (CI No. 201) is an excellent indicator for magnesium and has several advantages over Eriochrome Black T; Omega Chrome Red B (CI No. 652) can be used for the titration of Ni at pH 4.6 where relatively few ions interfere; Fast Sulphon Black F (CI No. 306) is specific in its indicator action for copper, and Acid Alizarin Black SN (CI No. 337) is particularly recommended for the titration of calcium in the presence of magnesium.

A new type of indicator synthesised from 1,2-dihydroxyanthraquinone-alizarin complexone—is superior to most others for the titration of Pb and Zn particularly, but is also recommended



for copper, indium and cobalt in acid solution. The cerous complex of this reagent gives a colour reaction with fluoride ion which can be used for the specific detection or determination of fluoride ion.

Symposium Members Discuss a New Microbiological Method

THE following is a summary of the discussion on the paper presented by Dr. D. J. D. Nicholas on the use of fungi to determine trace metals in biological materials (see CHEMICAL AGE, 30 August, p. 344).

Dr. J. Haslam (ICI, Plastics Division, Welwyn Garden City) thought Dr. Nicholas had been rather modest about his method and particularly in believing that modern chemical methods would prove more sensitive. Might not the truth be the other way round; that is, that sooner or later, the principle of the test which he (Nicholas) had pioneered, would be used by inorganic analysts to determine trace metals in inorganic salts. He gathered that in his method, the nutrient solution was taken, and the soil added. This was incubated and a mould growth was obtained. He wished to ask Dr. Nicholas two questions: (1) how was the mould growth assessed, and (2) why, with a given soil, does he not work at the pH of the particular soil he was examining?

Processing Mould Growth

Dr. Nicholas said it was possible to take the *Aspergillus* mould felt, wash away the soil and dry the mycelium at 99°C. This killed the fungus. It was then heated for 12 hours at 75°C in an oven, cooled, and this process was repeated until a constant weight was obtained. Reference to a standard was run as a matter of course. One could use three points or growth curve. The results should be within ± 3 to 5 per cent.

Molybdenum occurs in acid soils. One tended to adjust the pH to 7.5 for alkaline soils and manganese deficiency. Molybdenum was usually found in soils just about pH 5 to 6.5. The pH was adjusted to 5 to get some differential. If the pH of the culture solution was adjusted to pH 2, a very marked difference was noted. The amount of molybdenum required by the fungus was 0.001 μg . The fungus was very obliging as it grew between pH 1.8 to 7.5.

Mr. Simpson (London) said he gathered it was important to remove all trace

elements. He also wondered if Dr. Nicholas knew of any method for microbiological estimation of titanium.

Dr. Nicholas informed Mr. Simpson that it was not essential to remove all elements. Copper sulphate removes molybdenum but does not reduce iron. One only removed trace metal one is interested in. With use of copper sulphate, care had to be taken with copper and molybdenum. To trace the metal one wanted, all the trace metals were added back but the molybdenum was withheld.

With regard to a microbiological method of estimating titanium, Dr. Nicholas said he knew of no microbiological test for this element. He reminded Mr. Simpson that the element must be essential for the organism and not replaceable by any other.

Discussion on Dr. Levy's Paper

A point made by Dr. Al. Steyermark (Hoffmann-La Roche, US) during the discussion on Dr. R. Levy's paper (see CHEMICAL AGE 30 August, p. 346) was that he believed a source of low results in the Dumas method with compounds containing NCH_3 groups to be due to the formation of methylamine. The proof of this, he thought, was that these same substances would give correct results by the Kjeldahl method, but when the distillate was made alkaline, the odour of methylamine escaped Kjeldahlisation, it was conceivable that it escaped combustion and then was partially dissolved in the kof of the azotometer. Obviously, he stated, the formation of methylamine would not affect the results with the Kjeldahl method, since mol for mol, NH_3 and CH_3NH_2 would require the same amount of acid.

The exclusive 'Chemical Age' report of the International Microchemistry Symposium will be concluded in our next issue, with summaries of the papers by Dr. W. Schoniger (and discussion), the lecture by Dr. K. L. Cheng, and of the demonstrations that were held.

Overseas News

RECORD FIRST HALF-YEAR TURNOVER FOR WEST GERMAN CHEMICAL FIRMS

IN the first half of 1958, the West German chemical industry had the record turnover of DM 8,730 million (about £728 million). This was an increase of over 4 per cent on the total for the same period of 1957. However, the growth of the industry was slowing down; the 1957 half-yearly figure had been 14 per cent higher than that for 1956.

This was announced by the German Association of the Chemical Industry in Frankfurt-on-Main on 28 August. Despite the slower progress there were no signs of a recession in the industry; chemical manufacturers were at present benefiting from a demand for materials for plastics and artificial fibres greater than ever. It was pointed out that the increase in turnover for German industry as a whole had been only 3 per cent.

The chemical industry now had the third largest turnover in Germany. From the aspect of personnel, since the end of last year the number employed in the industry had risen by 7,000 to a total of 422,000.

Production stood at an index figure of 237 reckoned on monthly averages of 1958 so far (1938: 100). At this time last year the index had stood at 221. This steep increase had been due mainly to the increase in organic chemicals manufactured, particularly in the field of petrochemicals. The market in inorganic chemicals had reached more or less a saturation level with investments as they now stood.

Price indices were remarkable in that they had risen slowly compared with other industries. With 1938 levels as 100, this year's figure—which was dropping at present—was 189, only one point higher than last year. Prices in German industry as a whole had risen three points to 234 since last year.

US-French Detergent Tie-up

Formation of a new company, Soc. Petrosynthèse, is announced in France, following the US influence on French synthetic detergents. The company is a joint subsidiary of Oronte Chemical Co., San Francisco, California, US, and the French Cie Française de Raffinage (CFR), Paris, and Atlantique-Progil-Electrochimie (APEC), Paris.

Soc. Petrosynthèse will manufacture detergent alkylates. A plant is to be set up in the neighbourhood of Le Havre having a yearly capacity of 14,500 tons.

New £1 Million Plant Meets Mexico's Caustic Soda Needs

Mexico's total caustic soda requirements are now being met by the output of the Sosa de Mexico's new £1 million factory at Monterrey, which started

operations in June. In addition to 6,000 tons of caustic soda, the plant's annual production includes 5,000 tons of liquid chlorine, 2,000 tons of hydrochloric acid, 1,000 tons of sodium hypochlorite, and quantities of carbon tetrachloride and chloroform.

Total consumption of sulphuric acid in Mexico rose to 247,721 tons in 1957, only 737 tons short of domestic production. Ethyl alcohol production rose to 44,300 tons, but supplies still have to be imported.

Trinidad Fertiliser Plant Due on Stream Autumn 1959

Production is to begin at the fertiliser plant now being constructed by Federation Chemicals in Trinidad (see CHEMICAL AGE, 16 August p. 269) by August or September 1959.

A canal to the plant is now being dredged to enable shallow-depth boats to take materials to the site as well as take out fertiliser after the plant is in operation. This phase of the work is expected to be complete in two months' time.

Cost of the entire project when completed is expected to cost between \$20.4 million (W.I.) and \$22.1 million.

Dominica's Nationalised Nitric Acid Plant

The Government technological services of the Dominican Republic, which already operate a plant producing 12½ tons of sulphuric acid per day, have set up a RDS150,000 (£54,000) plant to produce five tons of nitric acid every 24 hours.

Dutch Technicians For S. African Urea Project

Technicians of the Dutch firm of Verenigde Machinefabrieken are on their way to supervise the building of a urea plant at Modderfontein, in the Union of South Africa. Urea-base artificial fertilisers will be produced at the plant by a process of the Dutch State-owned concern Staatsmijnen, one of Holland's most important fertiliser manufacturers.

Canadian Polythene Plants for Du Pont

Du Pont of Canada are to build a plant at Whitby, near Toronto, to produce polythene film, and the company's facilities at Shawinigan (Quebec), for production of cellophane cellulose film (KST polymer-coated film) are to be expanded.

Polythene film in sheet and tubing for the packing and construction industries is to be produced at Whitby. Construction work on the new plant is due to

start within 10 days. Work will also be started on a plant to make linear polythene resins near Sarnia (Ontario). This plant, the St. Clair River Works, will be of the automatic type and have open-air process building operated from a central control room. Raw material will be ethylene from Canadian Industries Ltd. new petrochemical plant in Sarnia.

Sulphuric Acid Output By African Explosives

Annual report of African Explosives and Chemical Industries shows that sulphuric acid output in South Africa now totals 420,000 short tons a year. The company's Rhodesian plant has a capacity of 67,000 tons a year.

Acrylonitrile Capacity Doubled

Production capacity for acrylonitrile has been doubled at the Institute, West Va., plant of Union Carbide Chemicals.

Pioneer Plant To Purify Water Pumped From Gold Mines

A pioneer plant to purify by electro-dialysis 2½ million gal. of brackish water a day is nearing completion in the Orange Free State gold field between Welkom and Adendaalsrus, where water pumped from the workings has become a vast artificial lake. The high salt content makes it unfit for agricultural or domestic purposes.

The new plant, forerunner of others to be built in the area, is based on research on electrodialysis started by CSIR early in 1953.

India Offered Russian Aid To Expand Pharmaceuticals

A team of eight Soviet experts have been discussing in New Delhi the further development and expansion of the manufacture of pharmaceutical products and drugs in India. The visit follows an offer made by the Soviet Union to provide financial and technical assistance for the development of this industry. The deal is said to include a loan of 80 million roubles (£3 million) for the purchase of machinery and equipment.

Yugoslav Fertiliser Plant

The superphosphate plant at Prahovo, Yugoslavia, planned for a capacity of 550,000 tons a year, is expected to have its trial run by the end of 1959 or at the latest during the spring of 1960.

Union Carbide Plan New Epoxide Unit In US

Union Carbide Chemicals Co., New York, US, are to proceed with an expanded version of a new unit at Institute, West Virginia, to produce more than 10 million lb. per year of epoxides and other oxygenated chemicals. This decision of expand operations show the company's confidence in a group of products which, it is said, have never before been available in commercial volume. The unit will be completed in mid-1959.

Among the new products are epoxide 201, (3,4-epoxy-6-methylcyclohexylmethyl-

3, 4-epoxy-6-methylcyclohexanecarboxylate), vinylcyclohexene dioxide, vinylcyclohexene monoxide, dicyclopentadiene dioxide, styrene oxide, allyl epoxystearate, and caprolactone. These products are expected to play an important part in resin technology and to serve as chemical intermediates as well. Special emphasis is to be placed on epoxide 201, which forms plastics and coating resins of outstanding colour stability and resistance to heat distortion. Epoxide 201 is claimed to be more reactive to acid and anhydride hardeners than any other diepoxide. In addition, Carbide will market two combination plasticiser-stabilisers for vinyl resins, di-2-ethylhexyl epoxy tetrahydrophthalate and di-isodecyl epoxy tetrahydrophthalate.

US Firm May Take Over Israeli State Fertiliser Co.

It is reported from Tel Aviv that the Israeli Government is planning to cede to a US company the State-controlled Fertilizers and Chemicals Ltd. of Haifa, the plant of which was purchased mostly in the United States at the cost of about 20 million dollars (£7 million).

German Chemical Interests In Argentina

The German Behring Group has purchased its expropriated Argentine subsidiary, the Instituto Behring de Terapeutica Experimental, EN, for the sum of pesos 17 million. No bid was made for the Quimica Bayer, EN, and the offer to sell was withdrawn. This firm will come up again for auction within 90 days at a 25 per cent reduction in the basic price.

High Temperature Silicon Compound

A report from Haynes Stellite Co., a division of Union Carbide Corporation, US, indicates that the company have produced a silicon compound that can resist temperatures over 3,000°F. The compound is described as silicon nitride and is produced by reacting elemental silicon metal with nitrogen. It is stated to retain its strength above 2,000°F and can be used in applications of low stress over 3,000°F. Resistance to most acids, including hydrochloric, nitric, sulphuric and phosphoric acids is claimed.

Cabot To Build Carbon Black Plant At Ravenna

Società Cabot Italiana of Rome, an affiliation of Cabot Laboratories of Boston, are to build at Ravenna a plant for the production of carbon black for the rubber industry. Production is scheduled to begin in about a year's time. Capacity will total about 14,000 tons a year.

New Dutch Company To Market Plastics Materials

A new company, the Vereenigd Plasticverkoopkantoor NV (the United Plastics Marketing Office) is to be set up in Holland by the Algemene Kunstzijde Unie, a large group of artificial silk and chemicals manufacturers, and the Dutch State Mines. The company will concern itself

with the selling of plastics both in the Netherlands and overseas. The company is to market Akulon products made by the Kunstzijde Unie as a basic nylon material for the plastics industry and polythene produced by the State Mines under the name of Stamylan.

A laboratory will be set up on the premises at Zeist of the new company at which research will be carried out into the potentialities of Akulon and polythenes.

Montecatini's Polythene Monopoly To Be Broken

Celene S.p.A. of Milan (a joint Sic-Edison and Carbon Chemicals Company concern) are building at Priolo Melillo, not far from Siracusa, Sicily, a plant for the production of polythene scheduled to go on stream in 1959.

So far, Società Montecatini have been the sole producers of polythene in Italy. Consumption last year was about 9,000 tons.

£4 Million Scheme To Work Uganda Phosphates

A £4 million scheme for exploiting the large phosphate deposits located at Sukulu, near Tororo, has recently been announced by the Uganda Development Corporation Ltd. It envisages the mining of over 2,500,000 tons annually to produce 400,000 tons of phosphate.

New Synthesis of Aliphatic Dinitriles and Diamines

Described in a recently issued patent to US Industrial Chemicals Co., New York (US Patent No. 2,824,118) is a new method of synthesising aliphatic dinitriles and diamines—some of which are believed to be new compositions of matter.

In the process isosebacic acid is reacted with ammonia and/or urea or other nitrogen containing compounds to form a mixture of C10 nitriles. These

are mainly sebaconitrile, α -ethylsuberonitrile and α,α' -diethyladiponitrile.

The nitrile mixture can be hydrolysed in the presence of ammonia and a catalyst to yield C₁₀ diamines. Those formed are a mixture of decamethylene diamine, 1,8-diamino 2-ethyloctane and 1,6-diamino-2,5-diethylhexane.

Suggested uses for dinitriles are as chemical intermediates, and for solvents, lubricants, plasticisers and textile water repellants. Applications for diamines would be in the synthesis of various types of polyamides—particularly fibres, and in plastics materials, rubbers, plasticisers, synthetic lubricants, polyurethanes and other products.

New Colombian Nylon Plant To Be Equipped By W. Germany

The Colombian firm of Vanylon Ltda., which is building a nylon plant with a monthly production capacity of 24,200 lb. at Barranquilla, Colombia, has ordered all its machines from the West German firm of Hans J. Zimmer, of Frankfurt-on-Main. The raw material to be used, caprolactam, will also be imported from the Federal Republic for use at Barranquilla until it can be produced in Colombia. The plant is the first of its kind in Colombia and will come into operation in mid-1959. Within a year it is expected that production of nylon will be increased fourfold. The country's demand is said to stand at well over 100,000 lb. a month.

US Merchants Acquire Glyco Products Co.

Chas. L. Huisking and Co., New York, merchants and producers of raw materials for the pharmaceutical industry, have bought the assets of Glyco Products Co. producers of chemicals for the food, plastics, paper, electronics, metals, paint and textile industries, and will form a new corporation, Glyco Chemicals, as a manufacturing and sales subsidiary.

New US Cyanosilicone Rubbers

GENERAL ELECTRIC of the US have introduced cyanosilicone rubbers, which although oil- and solvent-resistant, retain their rubber-like properties from 100°F to over 500°F. Basically the rubbers are methyl silicone rubbers having 10 to 50 per cent of their methyl groups modified to nitriloalkyls. The most basic type has one hydrogen in the methyl group replaced by a nitrile group to form a methylene nitrile (cyano-methyl) side chain $-\text{CH}_2\text{CN}$. Substitution by higher nitroalkyls, e.g. ethylene nitrile, produces variations in the characteristics of the finished rubber.

As the number of $-\text{CH}_2-$ groups between the nitrile radical and the silicon atom increases, the effect of the cyano group is stated to decrease. 'Repeat frequency' is described as important in the nitrile substitution, which can be altered by controlling starting materials

in the polymerisation. The polar nitrile group was chosen by General Electric researchers to reduce the affinity of silicone rubbers for non-polar hydrocarbons.

Three steps in the preparation of the compounds are chlorination, Grignard reaction, and polymerisation. A practical manufacturing process for the new material has been developed, General Electric report. High purity nitrile monomer is made, and is then hydrolysed and polymerised. Conventional techniques are applied to process the resulting long chain polymer. A filler such as silica is used together with a vulcanising agent, e.g. benzoyl peroxide and pigments if required.

A plant is now under construction at GE's Waterford plant, and the company hope to offer a standard product for sale late this year at an introductory price of about \$15 per lb.

● **MR. E. LORD, B.Sc.**, of the British Cotton Industry Research Association, Didsbury, Manchester 20, has been awarded the Textile Institute's Warner Medal, in 'recognition of outstanding work in textile science'. Mr. Lord, who joined BCIRA in 1930 as a laboratory assistant, has gained recognition for his work on statistical methods, particularly in connection with 'The use of range in place of standard deviation in the t-test'. His wife also works in the laboratories of the Shirley Institute.

● **MR. RAYMOND E. LAPEAN**, who for the past six years was managing director



R. E. Lapean

of Cyanamid of Great Britain Ltd., has been appointed regional director, Europe, of Cyanamid International, a unit of the American Cyanamid Co. He is to open a new European regional office at 48 Beethovenstrasse, Zurich, from which European marketing activities will be co-ordinated. Cyanamid, it is stated, do not intend to engage in basic chemical manufacturing in Europe, but they are seeking areas of demand for certain chemical specialties which are being developed.

● **MR. E. R. DE B. VARE, M.A.**, has been appointed general manager of Evans Medical (Wales) Ltd., Swansea, in succession to Mr. G. A. WILSON, who is taking up another appointment within the group. Mr. Vare, educated at Oundle and Magdalene College, Cambridge, joined the company in October 1954.

● **MR. W. H. GLASS, O.B.E.**, for 25 years a director of Thermotank Ltd., air conditioning engineers, of Helen Street, Glasgow, and a pioneer of modern air conditioning techniques, has resigned from the board at the age of 70. Installations designed by Mr. Glass included the air conditioning system in the operational headquarters built deep inside the Rock of Gibraltar, and that of the military hospital at Singapore.

● **DR. G. E. FOSTER**, chairman of the fifth British Pharmaceutical Conference, which will be held at Llandudno from 15 to 19 September is an organic research chemist on the staff of Burroughs Wellcome and Co. Theme of his opening address will be 'Modern analytical chemistry in the service of pharmacy and medicine'.

● **MR. C. P. PERCY**, chairman and managing director of J. M. Steel and Co. Ltd., London, and leader of the UK sea-going party to the meeting of the Society of Chemical Industry in Canada, sailed from Liverpool for Montreal on 5 September in the *Empress of Britain*. A fellow passenger attending the confer-

PEOPLE in the news

ence was Mr. C. L. EVANS, director of Brotherton and Co. Ltd., Leeds. Other chemical industry personalities aboard were: Mr. FRANK SMITH, works general manager, Associated Ethyl Co. Ltd., Ellesmere Port; Mr. J. DONALD BARR, joint managing director, ICI Paint Division, Slough; and Mr. JULIAN M. LEONARD, managing director, Carless Capel and Leonard Ltd., London E9, and a past-president of the SCI.

● **MR. J. T. M. DAVIES**, who has been manager and deputy manager respectively of the Swiss and Irish branches of ICI (Export) Ltd., has been appointed managing director of Imperial Chemical Industries (South Africa) in succession to Mr. G. E. HUGHES who has become managing director of African Explosives and Chemical Industries Ltd. Mr. Hughes who was appointed head of food and agriculture on the Allied Control Commission in Germany in 1946, returned in 1953 to ICI, whom he joined in 1928.

● **MR. A. P. M. PURDON** has been appointed operations study manager in charge of central operational research and work study for the British Oxygen Co. Ltd., Bridgewater House, Cleveland Row, London SW1.

Synthetic Resins Used In Uranium Extraction

IMPORTANT new chemical processes developed by the Australian Commonwealth Scientific and Industrial Research Organisation include what Mr. R. G. Casey, Minister in charge, describes as a revolutionary process for the production of uranium from the crude ore, which is about to be licensed on a world-wide basis.

Called the Weiss-Swinton process after its inventors, it is a method of controlling the separation process during which synthetic resins collect uranium on their surfaces so that it can be separated from the complex solution in which the crude ore is treated.

The basis of a process for the production of pure zirconium from Australian beach sands has recently been sold to a US company for £A110,000.

400 Chemists to Attend SCI Meeting in Canada

AN ATTENDANCE of 400 will mark the annual meeting of the Society of Chemical Industry to be held in Montreal on 14 and 15 September, and in Toronto on 17 and 18 September. Before and after the business sessions, visits will be made to plants in Quebec, Shawinigan, Sarnia, Niagara Falls and Ottawa.

During the Montreal meetings, the SCI Messel Medal will be presented to Mr. C. D. Howe, former Canadian Trade Minister for 'meritorious distinction in science, industry and public affairs.' At Toronto, the US section of the society will present its Chemistry Industry Medal to Mr. Fred. J. Emmerich, past chairman of Allied Chemical Corporation for 'conspicuous services to applied chemistry.'

Mr. H. Greville Smith, president of Canadian Industries Ltd., and president of the SCI is the first Canadian to hold the society's presidency in 20 years.

Agreement on Boron Isotope Production

A TEN-YEAR agreement has been signed by the UK Atomic Energy Authority with 20th Century Electronics, New Addington, Croydon, on the production and supply of stable isotope boron-10. The firm has transferred the low temperature distillation plant for producing boron-10 from the AEA Capenhurst factory to New Addington.

Training Courses In Polarography

FULL-TIME and part-time courses aimed at providing basic training in the theory and practice of polarography will be available during the session starting November 1958, states the British Polarographic Research Institute. Instrumental training will be given on manual, d.c. pen-recording, cathode ray, phase-selective and square wave polarographs.

Attendance on only one or two consecutive days a week will be necessary, but some private study will be desirable. Normal length of training will be 30 hours. Courses will run continuously from November. Further details are available from Mr. W. J. Parker, secretary, at 55 Oriental Road, Woking, Surrey.

BHC Butadiene Tanker in Road Accident

Prompt action prevented an explosion when a road tanker carrying butadiene was involved in an accident near Carlisle just before midnight on 6 September. The 3,600 gall. load started to leak and the driver stopped all passing traffic to avoid setting the gas alight from the spark of a car ignition. Staff from British Hydrocarbon Chemicals were on the scene at 6 a.m. with two empty tankers. Nitrogen was pumped into the damaged tanker to force out the butadiene, while firemen sprayed water to keep down the escaping gas. The road was cleared by 2.30 p.m.

Chemist's Bookshelf

ECONOMICS OF PLANT DESIGN

PLANT DESIGN AND ECONOMICS FOR CHEMICAL ENGINEERS, by Max S. Peters. McGraw Hill Book Co. Inc., New York. 1958. Pp. xi + 511.

The tone of this book is set by the frontispiece—a photograph of an ethylene dichloride unit—which is entitled $S + CH_2=CH_2 + Cl_2 \rightarrow CH_2ClCH_2Cl + S$. It is, as the preface states, an attempt to present economic and design principles as applied to chemical engineering processes and operations.

The first eight chapters comprising about one quarter of the text are devoted to economics of chemical processes, costs and cost estimating. These are followed by sections on general design consideration and optimisation of design together with separate treatment of important general cases such as waste disposal. The remainder of the text (chapters 13 to 17) is concerned with equipment design for materials handling, mass transfer, heat transfer and particle separation. These chapters review briefly the conventional methods of the design of equipment for the operations considered, and discuss side by side with this treatment the economic factors involved.

For this somewhat novel approach, wherein the elements of design procedure are presented at every step with an eye to their economic implications, the author is much to be commended. This synthesis of the practical and theoretical is most desirable at a time when the main emphasis in most undergraduate courses is on 'design from first principles', the first principles invariably being scientific ones. Where economics is included in

the course it often occupies a water-tight compartment, and is rarely adequately related to plant design. In reality, of course, economics is just as much one of the first principles of plant design as are the laws of physics and chemistry, and this book is to be welcomed for emphasising this, if for no other reason. Of course, the synthesis of what would have been so often regarded in the past as two differing approaches is by no means an easy matter, and in many sections the book shows a certain dualism.

In order to retain a manageable size of text, the author has been obliged to present design procedures in what often approximates to note form, and without derivations, although a fair number of references are given. This in itself is not necessarily a bad thing but one would have liked to have seen in the text more indication of the practical limitations of many of the design methods presented. A number of these are often only approximations, which in many practical cases become so complex that the simple economic calculations based upon them no longer hold.

The book ends with a number of well selected and graded problems, followed by tables which include average service life of equipment items, as well as the usual physical data information.

This volume is to be recommended particularly to the final year student or graduate apprentice chemical engineer. Once more it is a matter of regret that there is no comparable publication which relates to British costs and conditions.

D. C. FRESHWATER.

Advances in Mass Spectrometry

MASS SPECTROSCOPY, by H. E. Duckworth. Cambridge University Press. 1958. Pp. xvi + 206. 35s. net.

This recent addition to the Cambridge Monographs on Physics maintains the high standard of presentation and production associated with the series. The author has succeeded in giving an excellent account of the history of the development of mass spectroscopy from the early work of Aston until 1956-1957. A fairly detailed discussion of positive-ion optics is followed by chapters describing the sources that have been used for the production of positive ions and methods that have been devised for the detection of these ions. All the major advances in the development of mass spectrometers are included in the next two chapters dealing respectively with deflexion-type instruments and time-of-flight spectrometers.

Determination of isotopic abundances and of atomic masses are discussed in some detail and particular attention is paid to the sources of error in abundance determinations. The contributions of

mass spectroscopy in nuclear physics research are described in a separate chapter. It will perhaps be a little disappointing to chemical readers that little space has been devoted to the chemical applications of the mass spectrometer. These are discussed only briefly in the chapter on the ionisation and dissociation of molecules under electron impact. On the other hand, a fairly comprehensive account is included of the important applications of the technique in geology and, in particular, of the various methods for the determination of the age of mineralogical specimens.

The book is well written and well arranged. It includes an appendix setting out the abundances of the stable isotopes of the elements and their respective masses. The references are all given at the end of the volume in alphabetical order in the form of an author index. Although the book is primarily designed for physicists it will be of value to others concerned with mass spectroscopy, because it is so clear and readable.

C. KEMBALL

Data on Wide Range of Hydrocarbons and Sulphur Compounds

PHYSICAL AND AZEOTROPIC DATA. Hydrocarbons and Sulphur Compounds Boiling Below 200°C. By G. Claxton. The National Benzole and Allied Producers Association (NBA), London, 1958. Pp. v + 146. £2 2s, postage 1s 6d.

Prepared under the guidance of Mr. G. Claxton, chief chemist of Benzole Producers Ltd., this book should form a valuable work of reference to workers in many fields and a companion volume to *Working Data for the Benzole Industry*, which has already been published and to the new edition of *Motor Benzole, its Production and Uses*, the second edition of which was published in 1938. In preparing the third edition of *Motor Benzole* it was considered advisable to tabulate the more important properties of the hydrocarbons boiling below 200°C. As such a large number of these fall in this range, the NBA decided that publication of a separate book giving melting point, boiling point, density and refractive index of as many hydrocarbons as possible was warranted. Similar data for the sulphur compounds are included.

Information on azeotrope formation is also given. Due to consideration of only 14 main components, tabulation of the available information in a more usable form than has been produced elsewhere, has been possible.

Considerable care has been taken to give information which is accurate and up to date. Of marked value is the index which enables any compound to be traced from its customary name.

D.L.M.

Reference Tables for the Chemist

TABELLENBUCH: CHEMIE. Compiled by R. Kaltfen, I. Pagels and J. Ziemann, and edited by S. Otto and I. Pagels. Friedr. Vieweg, Braunschweig. 1958. Pp. x + 438. DM 17.60 (in German).

In this useful reference handbook there is a wealth of information arranged in over 50 tables of the type commonly consulted in the laboratory by physical, inorganic and organic chemists, especially those in industry. Tables are divided somewhat arbitrarily into three main groups: general, analytical and technical. The first group includes 17 tables, ranging from the periodic system and related tables concerned with atomic structure to gas solubility and dissociation constants of acids and bases, and includes a lengthy table of the properties of organic compounds (154 pages). The analytical group (13 tables) covers standard titration data, electrochemistry, etc., and the technical tables (21) give figures for such properties as specific heats, viscosity, specific resistance, etc. There are also a table of standard abbreviations, a literature index, a subject index and a useful table of five-figure logarithms.

The book is well produced on high-quality paper and has a pleasing linen binding. Its price is very reasonable.

A. R. PINDER.

RIC Lectures on 'Big Molecules'

BIG MOLECULES. By Sir Harry Melville, F.R.S. G. Bell and Sons Ltd., London. 1958. Pp. 180. 15s.

This excellent little book is based on a recent series of Royal Institution Christmas Lectures delivered by the author. Although it is designed primarily for young people with training in the basic principles of chemistry, there is much to interest the more mature student.

Great care has been taken to ensure simplicity of presentation; non-technical language is used whenever possible and there are many photographs and simple illustrations. This is not just another dull recital of theories and reactions, but an exciting adventure story of the exploration of new realms of chemistry and the harnessing of new materials in the service of mankind. The reader shares the excitement and fascination of the research laboratory and the challenge to the production engineer as new polymers are designed and developed for specific purposes in such diverse fields as textiles and tyres, dentures and food, flame-throwers and fireproofing, kitchen equipment and printed electrical circuits, medicine and warfare, jet aircraft and repairs to the Royal State Coach. This surely is the way to present modern science to young people if we are to ensure an adequate supply of enthusiastic scientists.

E. J. BOURNE.

50th Revision of Biltz Textbook

EXPERIMENTAL INTRODUCTION TO INORGANIC CHEMISTRY. By H. Biltz, W. Klemm and W. Fischer. Walter de Gruyter and Co., Berlin. 1958. Pp. 213 + viii. DM 14.80 (in German).

This is the 50th edition of the well-known book by Biltz which has been revised and brought up to date by the other authors. A short but useful introduction to laboratory technique is followed by sections which deal with simpler non-metallic compounds and with commoner metals and their compounds. Two further and more advanced sections are concerned with less common non-metallic and metallic compounds respectively.

Although emphasis is on experiment, theoretical interpretations and implications are inserted in some detail at appropriate points in the text. Specific reactions and characteristic tests are clearly indicated and there are a number of clear diagrams.

The teacher or student with a good knowledge of German will find the book of considerable value as a supplement to lectures of elementary and more advanced types and as an aid in the interpretation of the reactions of inorganic analysis, although group analysis tables are not included. Since the book is likely to find much use in student laboratories it is perhaps a pity that stronger covers could not be provided.

W. R. MOORE.

Chemist's Bookshelf

NEW EDITION OF FEIGL

SPOT TESTS IN INORGANIC ANALYSIS. By Fritz Feigl. Fifth English edition, translated by Ralph E. Oesper. Elsevier Publishing Co. and Cleaver-Hume Press, Ltd., London. 1958. Pp. xiii+600. 65s.

A new edition of 'Feigl' stands as a milestone upon the triumphal road of analytical chemistry. This self-contained volume of inorganic applications completes the final two-stage division of the text which was initiated two years ago by the appearance in its fifth edition of the independent volume on organic applications; a dichotomy which had become inevitable on account of the vast bulk of material to be handled. The enormously widened scope of spot-testing technique, for which Feigl himself has been largely responsible through his truly monumental researches, is reflected in the size of the present book in comparison with that of the first volume of the fourth edition—and in the very reasonable increase in price which it entails. To look back further to the third edition of 'Qualitative Analysis by Spot Tests,' published in 1947—a comparatively slim work of 574 pages covering both organic and inorganic radicals—is to clinch one's conviction that Professor Fritz Feigl, more than any other single figure in the world of chemical analysis, has made and continues to make history.

The first two chapters, which treat of the development of spot-testing techniques and working methods, remain much as they appeared in previous editions; for sheer simplicity, lucidity and

comprehension of their subject they deserve earnest recommendation to senior students in technical colleges, who will find them a worthy introduction to micro- and semi-micro technique and theory. The most voluminous increase in matter naturally affects the tests for metals, where the ever-widening availability of new and versatile organic reagents leaves its mark. There is an enlargement by nearly fifty per cent of the section on the application of spot-tests to the investigation of impurities and the routine examination of technical materials and minerals. The systematic qualitative examination of mixtures, where spot-testing can effect a great saving of time and material, is considered at length, and there is a detailed description of the separation of ions by means of the Weisz ring-oven. The interference of other ions is discussed, and the limits of detection set out throughout the book. At the end appears a tabulated alphabetical list of cations, anions and free elements with their appropriate reactions. The bibliography is remarkably comprehensive and manageable.

Here is treasure for the forensic investigator, the mineralogist and the pharmaceutical chemist, which they can ill afford to overlook. The new volume, with its flexible blue binding which is a joy to handle, is worthy to take its place in teaching, investigational and control laboratories alongside its green companion 'Spot Tests in Organic Analysis.'

PETER COOPER.

Problems of Nuclear Engineering

INTRODUCTION TO NUCLEAR ENGINEERING. By R. Stephenson. McGraw Hill Book Co. Inc., New York. 1958. 2nd Edition. Pp xi + 491. 74s.

A very good 'Introduction to Nuclear Engineering' has been produced by the revision of this book. The first edition was deficient in its lack of any consideration of the engineering problems associated with the reactor core beyond a review of materials. The new chapter on 'Reactor core design' adequately introduces the principal problems of heat transfer and mechanical design. Between editions, the author has also been able to draw on the information released at the first Geneva Conference and subsequently. Using this, he has particularly expanded the sections on materials and processing. There have also been many revisions to tables and diagrams.

The general pattern of the book has changed little. The introductory 'Review of nuclear physics' has been slightly enlarged. Then follows a chapter on

'Nuclear fission.' The third chapter on 'The nuclear chain reactor' has been rearranged to include some of the material of the first edition's second and fourth chapters. This rearrangement makes a better introduction to the reactors described. It is, however, disappointing to find that, although the description of reactors has been extended, Calder Hall is only mentioned in Table 3.11. The section on critical size studies has been enlarged in the next chapter on 'Reactor theory' and 'Radiation shielding' is very well treated in chapter 5 which now includes a section on 'Empirical equations for build up.'

Chapter 6 reviews 'Materials of Construction' and includes the effects of radiation on reactor materials. Brief mention is made of the difficult metallurgy of uranium. The very welcome addition of the chapter on 'Reactor core design' leads on from a general discussion of heat transfer to the problem

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of thermal failure in fuel elements due to the combination of a high thermal flux with a low coolant flow. This is followed by a discussion of mechanical factors and heat transfer media. A section is appended on 'Hardware' (pumps and so on) which formed part of the, now discarded, final chapter in the first edition.

There has been slight revision of the chapter on 'Reactor instrumentation and control.' Chapter 9 on 'The separation of stable isotopes' has still to find its most logical arrangement. It has been revised but not expanded; the brief section on accelerators has been rightly omitted. Chapter 10 has particularly profited by the Geneva releases. Under 'Chemical separation and processing' a succinct review is given of the chemistry of the heavy metals and

fission products together with a brief review of both aqueous and pyrometallurgical separation processes. It is a pity that the author found it necessary to omit description of crude uranium purification.

This second edition ends with a new chapter on 'Thermonuclear power' which is topical and quite interesting.

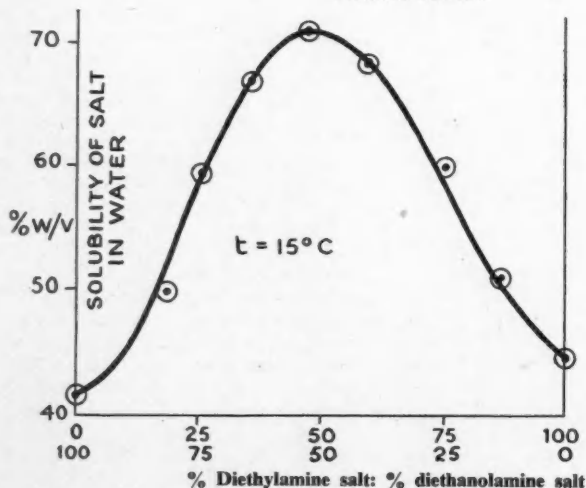
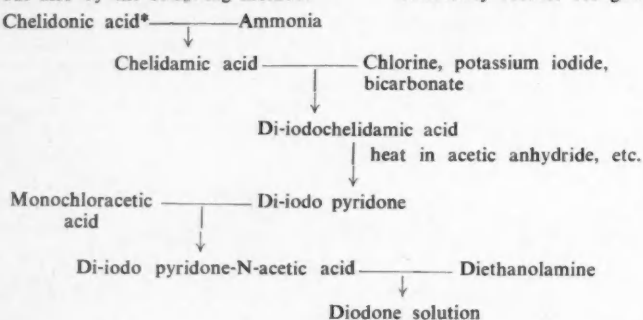
Not unnaturally, the book focuses on American achievements. Some may take exception to being referred to 'National data' on page 16. Perhaps the next edition will revert to 'Nuclear data.'

There are many more comprehensive texts on nuclear engineering but in fulfilling the aim of an 'Introduction' this book is well balanced and highly to be commended.

J. S. M. BOTTERILL.

Diodone 70 per cent A Stable Formulation—By Dr. M. A. Phillips

DIODONE B.P. is an aqueous solution of the diethanolamine salt of 3:5-di-iodo-4-pyridone-N-acetic acid; it is not in fact always manufactured by the method indicated in the British Pharmacopoeia 1958 (page 229) but also by the following method:



* From ethyl oxalate and acetone, *Organic Syntheses Coll. Vol. II*, p. 126.

be obtained. Indeed, it is sometimes considered, for some purposes, that the 35 per cent diiodone is not sufficiently concentrated and diiodone solutions containing 50 per cent of the salt and 70 per cent of the salt have been made, corresponding respectively to about 25 per cent and about 35 per cent iodine. They are administered by intravenous injection.

These stronger solutions tend to separate the salts, particularly in cooler weather and have to be warmed or heated by the clinician before use; in the case of the 50 per cent solution, this difficulty was overcome to a great extent by use of a mixture of the diethanolamine salt with a proportion of diethylamine salt (about 4 to 1), the latter conferring the expected extra solubility that might be expected from mixtures. Even so, this mixture, known by the proprietary name of 'Perabrodil forte' still tended to deposit crystals on cooling, even although it was better in this respect than the 50 per cent solution of the diethanolamine salt. To some degree, the problem of separation can be overcome by use of excess of the organic base giving solutions of high pH (about 8) but some objection can be raised to the use of alkaline injections.

It has been found that mixtures of the diethylamine salt with the diethanolamine salt of di-iodo-pyridone-N-acetic acid do in fact have solubilities in water which are greater than that of either component, the maximum solubility of which, at pH 7.2 are each about 30 per cent at 15°C . The maximum solubility is attained at the mixture of 51.7 per cent diethanolamine salt with 48.3 per cent of diethylamine salt, corresponding to one molecule of each and this solubility, at pH 7.2 and at 15°C , corresponds to about 34 per cent of combined iodine, that is to say, to the strength of Diodone B.P. 70 per cent. Such solutions do not deposit under ordinary winter conditions in this country and only to a small extent on chilling to 5°C . The deposit redissolves readily on warmth by the hand.

It has been suggested that these solutions cannot be employed on account of the higher toxicity of the diethylamine salt but it is considered that the evidence for this alleged higher toxicity is in fact not strong. More work on this toxicity is indicated since the advantages of using these stable strong solutions are manifest.

Humphreys and Glasgow Ltd. Are 66 Years Old

Humphreys and Glasgow Ltd., one of the three British firms concerned in the formation of the new consortium, Nuclear Chemical Plant Ltd., was founded in 1892, and not in 1951 as inadvertently stated in last week's issue (p. 397). Arrangements are being made for members of the company's scientific and technical staff to receive training with the UK AEA's Industrial Group

Commercial News

F. W. Berk and Co.

F. W. Berk and Co. Ltd. announce that the interim dividend on increased capital is 2 11/12 per cent compared with the corresponding interim of 2 11/12 per cent. Total dividend for last year was 10 per cent.

Elliott-Automation

Elliott-Automation Ltd., under an arrangement with Consolidated Electrodynamics Corporation of Pasadena, California, US, are to manufacture a wide range of analytical and control instruments of Consolidated design. The agreement is described by Elliott-Automation as an important step in broadening the range of equipment offered by the company. Negotiations for the deal have taken eight years. There is no financial tie-up and at the present stage additional capital will not be required.

Fisons in Canada

Fisons plan to extend their activities in Canada, a new company, Fisons (Canada) Ltd., having been formed to take over from the present distributors on 1 October. This has been announced by the chairman, Sir Clavering Fison. Although the new company will be primarily concerned with research and the distribution of British manufactured agricultural chemicals, production capacity in Canada may ultimately be developed.

It is considered that some of the firm's products to be marketed in Canada are technically superior to those of US competitors.

Smith and Nephew

Since Smith and Nephew Associated Companies merged with Southalls and Bertons, group turnover for the half-year to 30 June 1958, is some 5 per cent higher than for the same period of the previous year, including in both cases the sales of Southalls and Bertons.

Estimated profit, before tax, for the second quarter, is £510,000, making a total of £1,050,000 for the half-year. This represents a 14 per cent increase over the combined earnings of the three merged companies. Some £68,000 has to be set aside as preacquisition 1958 profits of Southalls and Bertons.

General indications are that the current half-year profit will be lower than the first.

United Glass Bottle

Interim dividend on ordinary is being maintained by United Glass Bottle Manufacturers at 3½ per cent.

Jefferson Lake Petrochemicals

Jefferson Lake Petrochemicals of Canada Ltd. report a loss of \$219,754 (£7,850) including write-off of \$107,752 (£3,850) for a dry hole in the Calgary, Alberta field, in the first six months of this year. Balance sheet showed working capital of \$2,444,185 (£873,000) including \$1,204,459 (£435,000) cash. In

- Berk Maintain Interim on Increased Capital
- Fisons Form New Canadian Company
- Jefferson Lake Plan New Sulphur Plants
- Naphtachimie Plan Large-Scale Expansion

the first six months the company produced and stockpiled 29,328 tons of sulphur pending completion of the Pacific Great Eastern Railway line to the Peace River plant site. The line is now completed and shipments have begun.

The report adds that the new Peace River plant is currently producing 160 long tons daily of high quality sulphur. In 1959, the plant will be able to produce over 300 long tons per day. Long range plans include a sulphur plant at Calgary and participation in a third plant at Savanna Creek, Alberta.

Reichhold Chemical (Aust.)

Profits of Reichhold Chemical Industries (Aust.) reached the record total of £A259,813 in the year to 30 June 1958, improving from £A245,349 for the previous financial year. Tax has taken £A136,540 (£A132,763) and depreciation £A100,763 (£A72,000). The dividend is being raised from 11 to 11½ per cent.

Operations in Victoria and New Zealand have been enlarged.

Naphtachimie

The French petrochemical company Naphtachimie S.A., founded in 1945 by Péchiney and Seichimé (Société d'Exploitations et d'Intérêts Chimiques et Métallurgiques) announce that in anticipation of the European Common Market they will launch another large-scale expansion programme. Already in recent years they have spent some millions of francs on expansion and new plant.

The investment of some F.Frs.15,000 million (£13 million) is envisaged. When the full programme has been completed by the latter part of 1960, Naphtachimie will be one of Europe's biggest petrochemical concerns. The new programme includes the installation of a cracking plant with a capacity of 32,000 tons of propylene and 30,000 tons of ethylene annually.

NEW COMPANIES

COMBUSTION CHEMICALS LTD. Cap. £100. Manufacturers of and dealers in combustion additives and fuel improvers, etc. Reg. office: 32 Bishopsgate, London EC2.

CHEMICAL EQUIPMENT ENGINEERING LTD. Cap. £2,000. Chemical plant design and manufacture, and engineers, etc. Directors: S. and P. M. A. Hopton, both of 255, Marsland Road, Sale, Ches, and D. S. Hopton.

SATISFACTION

GRIFFIN AND GEORGE (SALES) LTD. (formerly W. and J. George and Becker Ltd., and W. and J. George Ltd.) of Wembley, Middlesex. Satisfaction 4 July of deb. reg. 21 July 1952 (fully).

LONDON GAZETTE

Liquidator Appointed

ORGANIC INTERMEDIATES LTD., reg. office, 41 North John Street, Liverpool 2. E. Stevenson Browne, of the same address, appointed liquidator on 21 July.

Market Reports

BUYERS UNWILLING TO WORK AHEAD

LONDON There has been a quietly steady demand on home trade account for a general run of the soda and potash products, and most of the other routine industrial chemicals. Contract delivery specifications continue to cover good volumes but, as yet, buyers show little inclination to enter into fresh long-term commitments. A good flow of overseas enquiry has been reported, but the volume of orders placed for shipment has been moderate. Prices throughout the market are steady. Trading conditions in the coal-tar products market remain unchanged, with home demand keeping steady and export call rather slow except perhaps for refined tar and phenol crystals.

MANCHESTER Business in chemicals and allied products on the Manchester market is now pretty well free from seasonal influences and, on the whole, existing contracts are being drawn against

reasonably well. From the point of view of fresh bookings, however, home users tend to limit commitments to shorter periods than usual. Shipping business in most lines continues on a fair scale, though there has been no noticeable improvement in the demand for caustic soda and several other products which have been moving on a smaller scale than usual during recent months. A quietly steady demand for most of the tar products has been reported.

GLASGOW Although there is still room for improvement, quite an active week's trading can be reported from the Scottish market. Demands, on the whole, were fairly well maintained at nominal levels both against spot and contract requirements. Some sections did show a slight decline, but against this increases in other directions took place. Prices generally remained firm. The position in regard to agriculture still remains quiet.

TRADE NOTES

Bright Tin Plating Process

A tin plating process that will produce a bright deposit without polishing or scratch brushing has been developed by the Tin Research Institute and is now established on both a laboratory and production basis. The most important constituent of the bright tin plating process is the brightener, made from wood tar. A revivifying agent has also been developed for addition to an old bath when a decline takes place in the brightness of the deposits produced. The brightener and the revivifying agent, manufactured by Shirley Aldred and Co. Ltd., are available in England and Wales from Sonic Engineering and Equipment Ltd., 120-130 Parchmore Road, Thornton Heath, Surrey, under the name of 'Brytin', together with full technical service.

Permanent Instrument Exhibition

B and K Laboratories Ltd. have opened a permanent International Instrumentation Centre at their new premises, 4 Tilney Street, Park Lane, London W1. It will provide changing displays of new electronic developments, all-the-year-round instrument demonstrations, slide talks and films to extend application knowledge, an enlarged technical product library, and facilities for group discussion.

Mopump Branch Offices

To give better service in the Midlands and northern counties Rhodes, Brydon and Youatt Ltd., engineers and pump manufacturers, Gorsey Mount Street, Stockport, have opened two new registered area offices. Midlands area office (manager, Mr. C. M. Saunders), is at Griffin House, Ludgate Hill, Birmingham 3, tel: Central 8911/2. Northern counties office (manager, Mr. M. J. B. Hodgson) is at 60 Highbury, Jesmond, Newcastle upon Tyne 2, tel. 81:4722.

Change of Address

From 20 September the address of Cabot Carbon Ltd., will be 62 Brompton Road, London SW3 (Knightsbridge 7181).

C.N.S. Instruments Ltd.

The name of C.N.S. Instruments Ltd., manufacturers of electronic instruments, 61 Holmes Road, London NW5, was omitted from the current issue of the London A-D telephone directory, owing to a printing error. Readers are asked to note that the telephone number, Gulliver 2418, is unchanged.

Araldite Price Reductions

Substantially lower prices for Araldite epoxy resins have been announced by Ciba (ARL) Ltd. (formerly Aero Research Ltd.), Duxford, Cambridge. The reductions follow completion of a new epoxy resin factory at Duxford with a greatly increased production capacity.

Instrument Demonstration

The Beckman model IR5 double beam recording infra-red spectrophotometer is to be demonstrated by Mr. K. G. Sölla of Beckman Instruments in three sessions

at the London showrooms of Baird and Tatlock (London) Ltd., 14-17 St. Cross Street, Hatton Garden, EC1. The sessions will be held on 24 September at 2.30 p.m., and on 25 September, at 10.30 a.m. and 2.30 p.m. The IR5 has a range from 2 to 16 μ and a resolution of 0.03 μ at 10 μ . Visitors, who are asked to notify Mr. F. T. Hall of BTL at Freshwater Road, Chadwell Heath, Essex, of their intention to attend, may take samples for test if they bring their own cells.

First Aid Boxes

The First Aid in Factories Order prescribing the contents of first aid boxes is to be redrafted in the light of comments invited from industrial organisations by the Ministry of Labour. With the support of the British Employers' Federation, the TUC and the nationalised industries, the Ministry's factory inspectorate is to organise an increase in the number of trained first-aiders in factories.

DIARY DATES

MONDAY 15 SEPTEMBER

CS—London: Large Hall, Friends House, Euston Road, NW1., to Wednesday 17 September. Kekulé Symposium on Theoretical Organic Chemistry.

TUESDAY 16 SEPTEMBER

SAC—Birmingham: Mason Theatre, The University, Edmund Street, 6-30 p.m. Four papers by members of the Analytical Section, Research Dept., ICI Metals Division.

SCI, Corrosion Group, South Wales section of SCI and Cardiff section, RIC, joint meeting —Monmouth: Kings Head Hotel, Newport, 7 p.m. P. T. Gilbert, on 'Some factors affecting the performance of condenser and heat exchange tubes'.

Publications Received

Chemical Plant—From Design to Operation: Power-Gas Ltd., Stockton-on-Tees.

Organisation Overseas: George Kent Ltd., Luton, Bedfordshire.

Rigidex Rigid Polyethylene—Technical Information Sheets Nos. 3, 4 and 8: British Resin Products Ltd., Devonshire House, Piccadilly, London, W1.

Civil Engineering: Simon Carves Ltd., Cheadle Heath, Stockport.

Holmes-Connersville Exhausters and Boosters, Type XAS: Publication No. 75 of W. C. Holmes and Co. Ltd., Turnbridge, Huddersfield.

Reports on Plastics in the Tropics 8: Asbestos Filled Phenolic Resin: Ministry of Supply Report available from H.M. Stationery Office, Kingsway, London, WC2, price 4s. net.

Holmes Liquefied Petroleum Gas Installations: W. C. Holmes and Co. Ltd., Turnbridge, Huddersfield.

Telcon Audio Frequency Cables: Telegraph Construction and Maintenance Co. Ltd., Mercury House, Theobalds Road, London WC1.

Ciba Review, 127: Polyamide and Polyester Fibres. Deals with production of the polymers and modern trends in printing, dyeing and finishing. Ciba Ltd., Basle.

Index to Silicone Publications. (A0-10). Midland Silicones Ltd., 19 Upper Brooke Street, London W1.

Pyrethrum Facts: Press and Public Relations Ltd., 47-48 Berners Street, London W1.

Argonarc Equipment for New Chemstrand Plant

ALL PIPES at the new plant now being built for Chemstrand Ltd. at Coleraine, Northern Ireland, for the production of Acrilan fibre are of 18/8 stainless steel, the most of which is molybdenum bearing steel. Diameters range from $\frac{1}{2}$ in. nominal bore up to 18 in.

Welding is carried out on the site and pipework is hydraulically tested to 250 p.s.i. for the working pressure of 25-100 p.s.i. It is stated that so far no leakages have been detected and that only 4 per cent of welds have needed repair. Argonarc equipment was supplied to a welding shop on the site by British Oxygen Gases Ltd.

All pipes are purged internally with argon gas to prevent oxidation and before use they will be washed with acid solution and degreased.

Building of the plant will probably be completed by the end of this year; part of it is already in operation.

Antioxidants Permitted as Food Preservatives

FIVE antioxidants or their admixtures may be used within prescribed limits as preservatives of home produced or imported foodstuffs under The Antioxidants In Food Regulations, 1958, issued jointly by the Ministry of Agriculture, Fisheries and Food and the Ministry of Health. They are propyl, octyl or dodecyl galates, or butylated hydroxyanisole or butylated hydroxytoluene.

The use of such antioxidants in oils and fats, essential oils and butter for manufacturing purposes and in compound foods containing these substances was recommended by the Food Standard Committee.

Research Worker May Have Been Sensitive to Benzene

DURING a Manchester inquest on Patrick Freeman (24), research chemist employed by the Geigy Co. Ltd. at Rhodes, Manchester, held on 4 September, it was stated that nobody could find out in advance if a person was hypersensitive to benzene. Freeman died from a blood disease after exposure to the chemical in a laboratory where he had worked.

Dr. G. Taylor, pathologist, said some people could work with benzene all their lives without any ill effects, but others would be hypersensitive to the chemical. In that particular case he could not eliminate benzene as the cause of the disease.

A verdict of accidental death was returned.

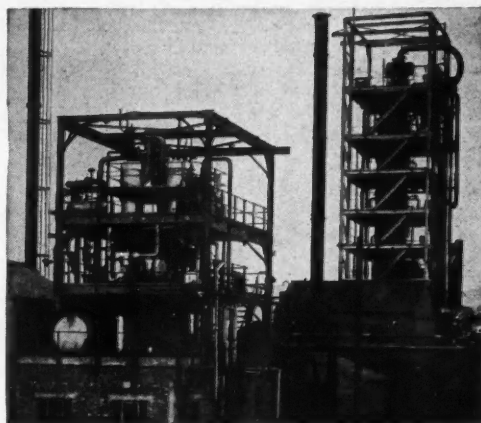
Vacuum Furnaces for AEA

Wild-Barfield Electric Furnaces Ltd., of Watford, have received an order from the Atomic Energy Authority, Risley, for two 100 lb. arc melting vacuum furnaces of the cold mould type. They will be used for consumable and non-consumable melting. Output is such that a 4 in. ingot can be melted in each furnace simultaneously with a 50 per cent reserve of power.

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ARMEEN 12D	pure lauryl	Intermediate for textile chemicals, quaternaries.	Armeens, L-12
ARMEEN T	technical oleyl, stearyl	Wax emulsions.	F-2
ARMEEN HT	technical stearyl, palmityl	Waterproofing, flotation.	Armeens, L-12
ARMEEN HTD	pure stearyl, palmityl	Hard rubber mould release, bitumen additive.	L-3
ARMEEN 18D	pure stearyl	Rubber industries.	Armeens, L-2
ARMEEN C	pure lauryl, myristyl	Fertilizer industry, anti-static.	G-3, G-5
		Boiler condensate control, intermediate for textile chemicals.	L-7
		Petroleum industry, corrosion inhibition.	
		Pigment manufacture, paint industry.	
★ Secondary Amines R_2NH			
ARMEEN 2C	technical lauryl, myristyl	For manufacture of quaternaries soluble in oils, etc., dispersible in water.	Armeens
ARMEEN 2HT	technical stearyl	Corrosion inhibition.	Arquads
★ Diamines $R.NH.C_3H_6NH_2$			
DUOMEEN T	technical oleyl, stearyl	Road making.	C-2/3
DUOMEEN CD	pure lauryl, myristyl	Paper, Waterproofing, surface coatings.	Duomeens
DUOMEEN T DIOLATE	technical oleyl, stearyl	Epoxy resins.	C-3/L/I
		Corrosion inhibition.	L-15
		Metal working, wire drawing.	G-4, L-14
		Paints, caulking compounds, pigments.	L-4, L-8
Water Soluble Quaternaries $[R.(CH_3)_3N]^+ Cl^-$			
ARQUAD 12	pure lauryl	Foam rubber.	L-5
ARQUAD 18	pure stearyl	Emulsifier.	
ARQUAD C	technical lauryl, myristyl	Anti-static for plastic.	L-11
		Germicide, cosmetics, road emulsions.	E-5
		Textile industry.	G-2
Oil Soluble Water Dispersible Quaternaries $[R_2(CH_3)_2N]^+ Cl^-$			
ARQUAD 2C	technical lauryl, myristyl	Corrosion inhibitor, petroleum production.	E-3, E-6, G-3
		Internal anti-static, germicide.	E-5
		Insecticide and other emulsifiers.	E-4, F-2
		Slip aid in transparent viscose films, etc.	
ARQUAD 2HT	pure stearyl, palmityl	Laundry & textile softener.	E-L
		Starch industry.	E-8
		Paper industry.	E-I

★ Each of these is also available in its water-soluble acetate form: ask for booklet on ARMACS

ARMOUR CHEMICAL INDUSTRIES LTD., 4 Chiswell Street, Finsbury Square, London, E.C.1.

Tel: METropolitan 0031

C5.



NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sale Branch), 25 Southampton Buildings, Chancery Lane, London WC2, price 3s 3d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 15 October

Compositions for combating nematodes. Farbenfabriken Bayer AG. **802 882**
Process for rectifying ethanol by distillation under elevated pressure. Chematur, A.B. **803 047**
Niobium-base alloys. Du Pont De Nemours & Co., E. I. **802 857**
Process for cracking hydrocarbons and a combination fluid solids reactor and thermal cracking unit. Esso Research & Engineering Co. **803 104**
Refining alcohols. Standard Oil Co. **802 860**
Halogen-containing synthetic resin compositions and a method of stabilising such compositions. Carlisle Chemical Works, Inc. [Divided out of 803 081.] **803 082**
Producing a tank surface for glass manufacture. Vereinigte Grossalmeroder Thonwerke. **802 950**
Bacteriostatic compositions. Scientific Oil Compounding Co., Inc. **803 080**
Dicyclohexyl alkyl phenols. Dow Chemical Co. **802 884**
Continuous process for the esterification of cellulose in homogeneous phase. Soc. Rhodiacta. **802 863**
Process for the production of N¹-substituted N-aryl sulphonyl ureas. Geigy AG, J. R. **802 885**

AMENDED SPECIFICATIONS PUBLISHED On Sale 8 October or as soon as possible thereafter

Herbicide compositions. Monsanto Chemical Co. **664 942**
Aminophenoxyalkane derivatives. Wellcome Foundation, Ltd. **770 411**
Cation-exchange resins. Permutit Co., Ltd. **781 780**
Polymerisation catalyst. Polymer Corporation, Ltd. **789 970**

Open to public inspection 22 October

Production of uranium halides. Murphree, E. V. **803 258**
Gasification of solid carbonaceous material in suspension. Gas Council, West Midlands Gas Board, Dent, F. J., and Stuart, R. T. **803 234**
Process and products for the improvement of the optical properties of organic material. Ciba Ltd. **803 361**
Imidazolium compounds and process for their manufacture. Ciba Ltd. **803 362**
Preparing 3-methyl-3-carboxycyclohexanone-(1)-2 β -propionic acid and derivatives thereof. Abildgaard, K. (trading as Lovens Kemiske Fabrik Ved A. Kongsted). **803 426**
Treating hydrocarbons in the presence of hydrogen and in producing alumina for use as a carrier. Badische Anilin- & Soda-Fabrik G.m.b.H. **803 261**

Producing fast dyeings on animal or synthetic fibres or mixtures thereof. Farbwerke Hoechst AG. **803 287**
Substituted acetone dicarboxylic acid compounds and production of an aliphatic keto-alcohol therefrom. Pfizer & Co., Inc., C. **803 164**
Preparing an alkali metal-titanium fluoride. Republic Steel Corp. **803 427**
Polymerisation of organopolysiloxanes. General Electric Co. **803 288**
Method of preparing mixtures of pseudo-irones. Givaudan & Cie. S.A., L. **803 364**
Organopolysiloxanes having pressure-sensitive adhesive properties. General Electric Co. **803 262**
Pharmaceutical compositions. Imperial Chemical Industries Ltd. **803 289**
Production of water or hydrogen having an increased deuterium content. Becker, E. W. A. **803 274**
Production of nitric acid. Imperial Chemical Industries, Ltd. **803 211**
Apparatus for contacting liquids with vapours. Imperial Chemical Industries, Ltd. [Cognate application 26 962] **803 212**
Methods of condensing metal halide vapours. Du Pont de Nemours & Co., Ltd., E. I. **803 432**
Coated regenerated cellulose film. Du Pont de Nemours & Co., E. I. **803 191**
Manufacture of granular materials, e.g., fertilisers. Fisons, Ltd. **803 437**
Extraction of metals from aqueous solution. UK Atomic Energy Authority. **803 264**
Oestradiol derivatives. Laboratoires Francais de Chimiotherapie. **803 440**
Stabilised polymers. Goodrich Co., B. F. **803 231**
Thiophosphoric acid esters. Farbenfabriken Bayer A.G. **803 441**
Process of bonding glass articles and compositions for use therein. Monsanto Chemical Co. **803 369**
Process for manufacturing sintered magnesia. Terni Societe per L'Industria e L'Eletticità. **803 358**
Method and apparatus for effecting liquid phase chemical reactions. Chematur A. B., and Norsk Sprængstoffindustri A.S. **803 370**
Removal of organic sulphur from industrial gases. Scientific & Industrial Research, Council of. **803 399**
Production of ammonium sulphate having a reduced tendency to setting. Imperial Chemical Industries, Ltd. **803 192**
Halobenzyl esters of thiophosphoric acid and their production. Farbenfabriken Bayer A.G. **803 446**
Purification of phthalonitriles and/or phthalimide. Distillers Co., Ltd. **803 172**
Preparation of thyroxine analogues. Glaxo Laboratories, Ltd. [Addition to 652 207.] **803 149**
Thioxanthene derivatives. Merck & Co., Inc. **803 401**
Organosilicon dielectric materials. General Electric Co. **803 150**
Organic nitrogen compounds and process for producing same. Parke, Davis & Co. **803 403**
Removal of fume from gases. British Oxygen Co., Ltd. **803 177**
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